DEVELOPMENT OF SUSTAINABLE CONCRETE SET RETARDERS USING INDIGENOUS INDUSTRIAL BYPRODUCTS

BY

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Dedicated to My Parents

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Firstly, all praise is due to Allah for granting me knowledge, strength and patience to fulfill this research successfully.

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LIST OF ABBREVIATIONS

ASTM	:	American Society for Testing and Materials
°C	:	Degree Celsius
CA	:	Coarse Aggregate
EAFD	:	Electric Arc Furnace Dust
FA	:	Fine Aggregate
FST	:	Final Setting Time
IST	:	Initial Setting Time
LSP	:	Limestone Powder
LVDT	:	Linear Variable Differential Transducers
MPa	:	Mega Pascal (N/mm^2)
OPC	:	Ordinary Portland Cement
SEM	:	Scanning Electron Microscopy
w/c	:	Water/cement ratio
Wt.%	:	Weight percent

ABSTRACT

Full Name : Osama Ghanem Rashed Massarweh

Thesis Title : Development of Sustainable Concrete Set Retarders Using Indigenous Industrial Byproducts

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Elevated temperature accelerates the rate of cement hydration and decreases the setting time of concrete. Retarding admixtures are generally used to increase the setting time, which reduces the accelerating effect of hot weather on concrete setting and provides a longer time for its transportation and handling. Though several commercial retarders are available to prolong the setting time of concrete, they have a shorter shelf life since they are in the form of a liquid. The use of expired retarders can cause delayed setting leading to a poor quality of hardened concrete. This deficiency brought about the need to develop solid retarders that can remain intact for a long period of time. The development of reliable retarders using indigenous industrial waste materials will be particularly useful. In this study, indigenous waste materials, such as electric arc furnace dust (EAFD) and oil ash were considered to develop solid retarders.

The retardation efficiency of the selected industrial byproducts was evaluated and compared with other commercial retarding admixtures along with other retarding materials including sodium gluconate, sugar and zinc oxide. Different dosages were selected based on setting time trials. The effect of the selected dosages on heat of hydration, slump

retention, compressive strength, drying shrinkage and morphological characteristics was examined.

The retarders developed using the selected indigenous industrial waste materials were efficient in enhancing the setting time. The use of retarders did not affect the properties of hardened concrete. Further, the performance of the developed solid retarders was similar to that of two selected commercial and other retarders, such as sugar, sodium gluconate and zinc oxide. Based on experimental data, dosages of retarders for the desired setting time and slump retention are provided.

ملخص الرسالة

الاسم الكامل: اسامه غانم راشد مصاروه

عنوان الرسالة: تطويرأضافات تأخير الشك المستدامة للخرسانة باستخدام مخلفات صناعية محلية

التخصص: الهندسة المدنية (الإنشائية)

تاريخ الدرجة العلمية: يوليو 2018

درجات الحرارة المرتفعة تزيد من سرعة تفاعل الا سمنت و تقلل مدة زمن الـ شك للخر سانة. إ ضافات تأخير الـ شك تستعمل بشكل عام لزيادة مدة زمن الشك مما يقلل من التأثير المتسارع للطقس الحار على تصلب الخر سانة و يوفر وقتاً اطول لنقلها و التعامل معها. رغم توفر العديد من إ ضافات تأخير زمن الـ شك التجارية والتي تطيل زمن الـ شك للخر سانة، الا أنها تمتلك فترة صلاحيةٍ أقصر لأنها تأتي على شكلِ سائل. إن ا ستخدام إضافات تأخير الشك منتهية الـ صلاحية يؤدي الى زمنِ شكٍ مؤجلٍ و من ثمَّ الى خر سانةٍ مت صلبةٍ ذاتِ نوعيةٍ رديئةٍ. وقد ادى هذا العيب لجلب الحاجة لتطوير إضافاتِ تأخيرِ الشك ذات القوام الصلب التي تبقى سليمة لفترة طويلة من الزمن. إن تطوير إضافات تأخيرِ الشك الموثوقة باستخدام موادِّ مخلفاتٍ صناعية محلية سيكون مفيداً بشكل خاص. في الدراسة المقترحة، مخلفات صناعية محلية مثل (EAFD) و (Oil Ash) تم أخذها بعين الاعتبار.

لقد تم اختبارُ كفاءة المخلفات الصناعية المختارة كإضافات لتأخير زمن الشك و مقارنتُها مع ا ضافات تجارية لتأخير الـ شك بالإ ضافة لمواد تأخير كيميائية أخرى بما في ذلك الـ سكر و أك سيد الزنك و جلوكونات الـ صوديوم. العديد من الجرعات تم اختيارها بالاعتماد على تجارب زمن الـ شك. تأثيرات الجرعات المختارة على حرارة الاماهة والحفاظ على التشغيل و قوة تحمل الضغط و الانكماش الناتج عن الجفاف والخصائص المورفولوجية تم اختبارها.

إضافات التأخير الشك التي تم تطويرها كانت فعالة في زيادة زمن الشك دون التأثير على خصائص الخرسانة المتصلبة. كذلك فإن اداء اضافات تأخير الشك التي تم تطويرها كان مشابها لاداء إضافات تأخير الشك التجارية و الاضافات الاخرى كالسكر و جلوكونات الصوديوم واكسيد الزنك. بالاعتماد على النتائج التجريبية تم تحديد جرعات الاضافات للحصول على زمن الشك المطلوب والمدة التشغيلية المطلوبة للخرسانة.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Concrete is one the most largely used construction material around the globe [1]. Primarily, it consists of fine aggregates, coarse aggregates, cement (binder) and water. With the enormous use of concrete, several challenges arose and led to imposing limits on its properties in both fresh and hardened states. According to Neville [2], during the fresh state, concrete mixes must be workable enough to be transported, placed and molded properly. One of the main requirements is that the consistency of a fresh mix should be adequate for consolidation to the maximum density without any excessive effort. In addition, the mix needs to be cohesive enough to resist segregation and be homogeneous during transporting and placing. Therefore, even though the quality of cement might be assured by the manufacturer, the final structural member and its quality is extremely dependent on concrete making and placing.

The quality of concrete is also affected by the hot weather conditions that lead to an acceleration in the progression of the hydration reaction or the rate of loss of moisture in concrete. Hot weather may include high atmospheric or concrete temperature, high wind speed or low relative humidity [3]. The Arabian Gulf area does not have a uniform climate

all over it. Generally, the area can be distinguished by two climate zones. The inland zone is hot and dry, and the coastal zone is hot and humid (Figure 1.1) [4].

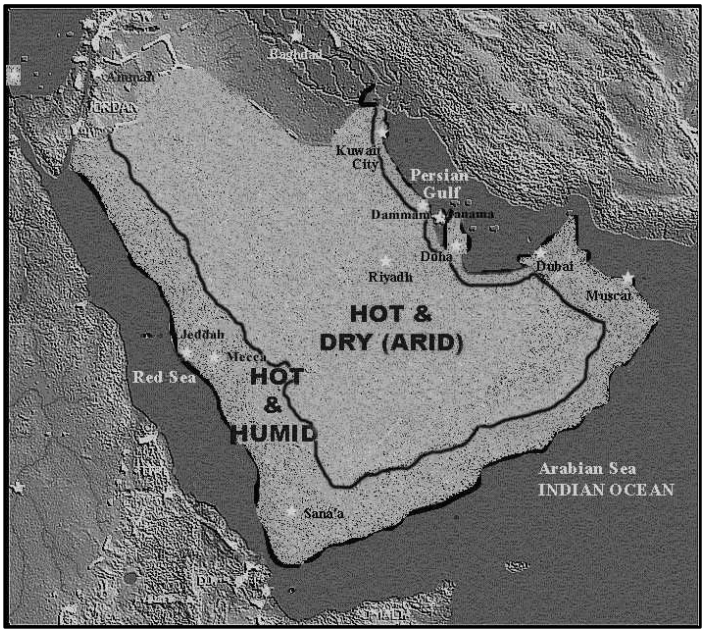

Figure 1.1: The Arabian Peninsula Climate Zones [4].

Hot weather causes a reduction in the setting time of concrete. It also causes problems in transporting and casting of concrete since it reduces the workability of the fresh concrete that results in accelerated rate of slump loss. During casting at higher temperatures, in some parts of the Arabian Gulf region, water may be added to fresh concrete to make it more workable and to increase the time of set which may cause plastic cracks that may impair the bond strength between concrete and reinforcing bars [5]. Plastic shrinkage cracks allow moisture and chloride to penetrate to the reinforcing steel thereby initiating reinforcement corrosion. Tests on cement mortars, Figure 1.2, indicates that the setting time was approximately reduced to the half upon raising the temperature of the mortars from about 27 to 45 °C [6].

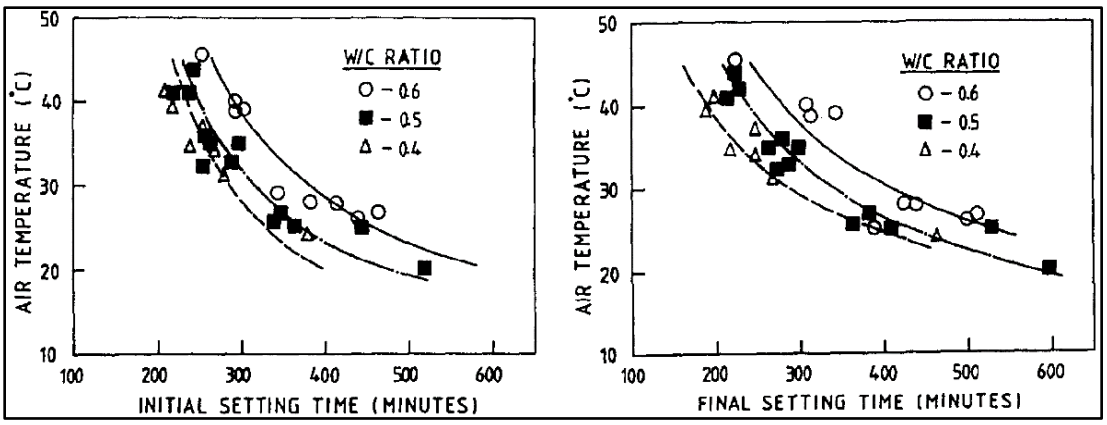

Figure 1.2: Temperature Effect on Setting time of Mortars [6].

Escalante-Garcia and Sharp [7] reported that the elevated hydration temperature raises the rate of hydration of the four main anhydrous mineral phases in cement. Kjellsen et al. [8] highlighted that raising the hydration temperature of cement paste leaves inadequate time for a proper diffusion of the hydration reaction products. Consequently, larger pores form, and the compressive strength is reduced. Price [9] showed that elevated temperature during the first two hours after concrete casting cause a reduction in its ultimate compressive strength (Figure 1.3).

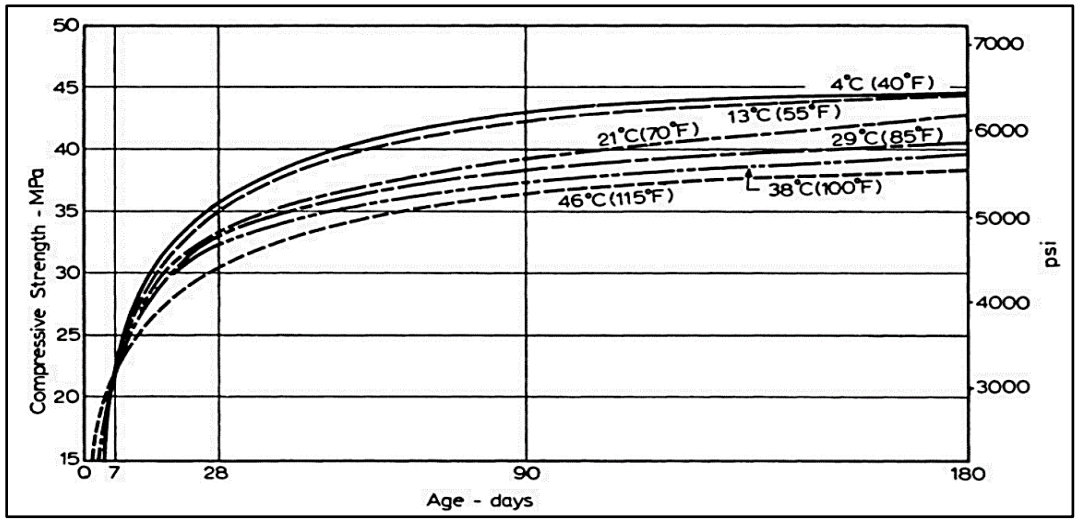

Figure 1.3: Influence of Higher Temperature in the First 2 Hours Following Casting on Concrete Strength [9].

High curing temperature maintained for 28 days also reduces the long-term compressive strength of concrete. Figure 1.4 presents the relationship between the age and the compressive strength at different maintained temperatures [10].

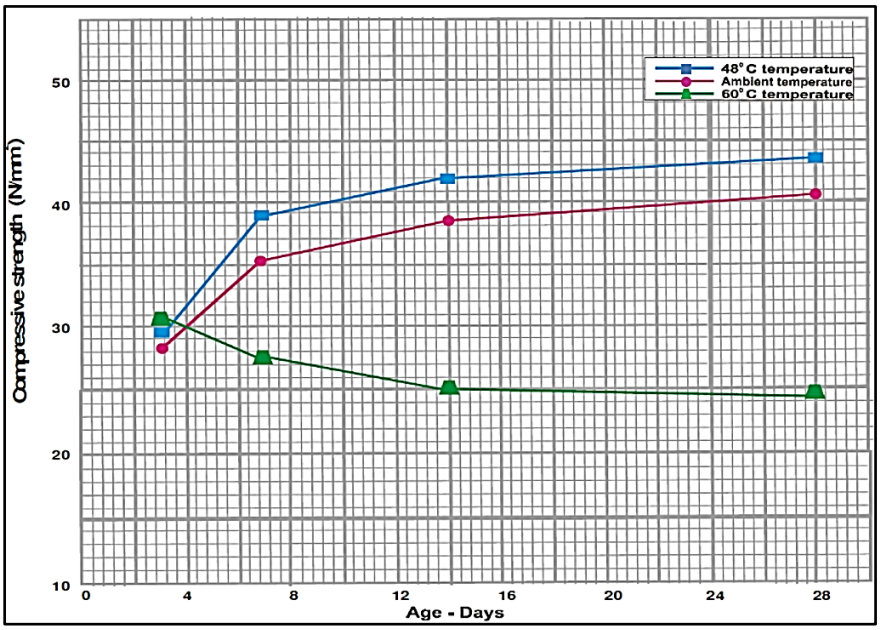

Figure 1.4: Curing Temperature's Impact on Concrete Strength [10].

The use of set retarders will temporarily hamper the setting of concrete. This will provide enough time for a longer transporting and a proper casting and finishing of fresh concrete. Thus, hardened concrete with appropriate properties will be produced. Set retarders prolong the concrete's setting time and reduce the quantity of water in the mixture; they also slightly increase the ultimate strength of concrete. Nevertheless, set retarders tend to decrease the early-age strength and may increase the hydraulic shrinkage [11].

Retarders decrease the rate of reaction between water and cement. They may also hinder the penetration of water to cement grains. The setting of normal-composition cement is related primarily with the hydration of C_3S and the generation of C-S-H [12]. The

mechanism of action of set retarders is hypothesized to be a poisoning to the morphology or crystal growth leading to a decrease in the growth of calcium hydroxide crystals [13].

Currently, a wide range of chemicals and additives are used for workability retention and retardation of setting time. Gypsum, common sugar, lignosulfonic acid are commonly used retarders [14]. Set retarders are classified into two groups. The first group includes the organic retarders, such as sugar, phosphonates and lignosulfonates (sulfonated lignin). The other group includes: the inorganic retarders, such as borates and zinc oxide. Organic retarders are more commonly used since they are less expensive [15]. Examples of chemicals used as the main constituent for commercially available retarders include: sodium gluconate, sucrose, glucose, citric acid, potassium biphosphate and zinc oxide. However, the commercial retarders are supplied usually in the form of liquid and they have a limited shelf life. In fact, an extended retardation of concrete has been noted due to the use of expired retarders. Hence, development of a retarder that has a prolonged shelf life is imperative. Solid indigenous industrial byproducts maybe possible candidates to be used as set retarders since many of them contains the ingredients that have retardation effects.

1.2 NEED FOR THIS RESEARCH

In modern times, concrete is a dominant construction material in engineered structures due to the wide availability of the concrete-constituting materials, such as gravel, sand, cement and water. Concrete also proved to be a versatile and competent construction material in respect of its strength, stiffness, durability, cost and ease of manufacture and placing in most conditions. However, hot weather poses many challenges on concrete work. In hot weather conditions, concrete begins to lose its workability rapidly during transportation

and placement phases. This creates concrete with inadequate capability of being compacted. Consequently, honeycombs, air voids and placement lines will appear. Thus, the hardened concrete will undergo a higher permeability, a lower compressive strength and a reduced durability. Furthermore, the long-term properties of hardened concrete are significantly affected by the setting time. After mixing, a reduced setting time due to higher temperatures leads to a dubious quality of concrete since any disturbance occurring after the initial setting can cause a breakup of the early-age cement hydration products. Sources of disturbance may include transporting, placing, handling and finishing operations.

In accordance with the above, concrete set retarders are of paramount importance to maintain and improve the workability of fresh concrete in the hot and harsh environments. Set retarders are also necessary to decrease the rate of hydration reaction, which may leave an adequate time for a proper formation of cement hydration products. Despite their continued use, the commercially available retarding admixtures are expensive and have a certain length of time for which they remain usable and fit for use.

Solid retarding agents developed from indigenous industrial byproducts can be considered a justifiable substitute for the conventional liquid commercial retarders due to economic and technical benefits. Solid retarders will remain intact for a much longer time than the liquid commercial retarders. Accordingly, this study was conducted to develop set retarders from industrial byproducts through the selection of the most suitable materials and dosages without jeopardizing the mechanical and other characteristics of the produced hardened concrete.

The use of indigenous waste byproducts in concrete is consistent with the general trend towards a reduction in greenhouse gas emissions. Moreover, concrete can act as a medium to accommodate part of the hazardous byproducts, such as EAFD and oil ash, thus, minimizing the environmental impact.

1.3 OBJECTIVES

The main objective of this study was to develop sustainable concrete set retarders using indigenous industrial byproducts, namely EAFD and oil ash.

The specific objectives were the following:

i. Collect samples of EAFD and oil ash,
ii. Assess the chemical composition of the selected materials,
iii. Ascertain setting time and slump retention of cement paste/concrete specimens prepared with varying dosages of the selected materials,
iv. Ascertain the mechanical properties of concrete containing the developed retarders,
v. Assess effects of the developed retarders on heat of hydration and morphology,
vi. Compare the performance of the developed retarders with that of commercial retarders, and
vii. Provide recommendation on dosages of the developed retarders.

1.4 APPROACH

A macro-level flowchart of the approach followed to achieve the objectives of this research is represented in Figure 1.5.

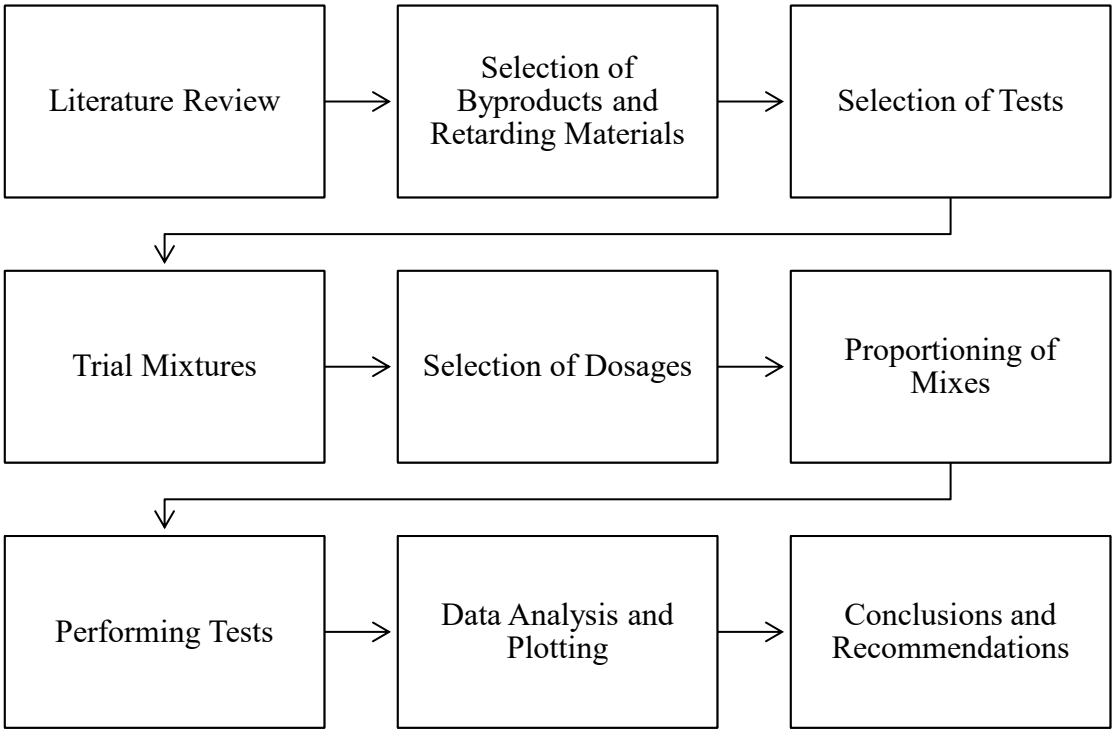

Figure 1.5: Flowchart of Approach

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

To achieve a delay in the setting time of cement, a retarding admixture or ASTM Type B chemical admixture can be added to the mix [16] . The mechanism of action of set retarders is hypothesized to be a poisoning to the morphology or crystal growth leading to a decrease in the growth of calcium hydroxide crystals [13]. The retarding admixture creates an effective barrier hindering further hydration until the retarder is incorporated into the structure of the hydrated material. Thus, the admixture is removed from the solution. However, that does not imply a formation of other complex hydrates [17].

This literature review delves into earlier research conducted to study the use of set retarders in concrete. It consists of three main parts. The first part presents common materials having retarding properties. The second part discusses the effects of the proposed industrial byproducts on setting time and other properties. Finally, the third part describes the behavior of concrete as affected by set retarders in hot weather conditions.

2.2 MATERIALS WITH RETARDING PROPERTIES

This section provides a review of common chemicals that have retardation effects. Thus, three chemicals were selected for use in this research. The economic feasibility of the

developed retarders (industrial byproducts) will be assessed based on a comparative study of the different materials considered.

2.2.1 Sugar

Sugar, which a robust common retarder, was examined by Bermudez [18]. The research was accomplished on cement slurries. The thickening time of the tested cement slurries depends mainly on the sugar concentration. Sugar needs to be added in a small concentration to act as a retarder. Figure 2.1 illustrates the effect of sugar on the thickening time at different sugar additions.

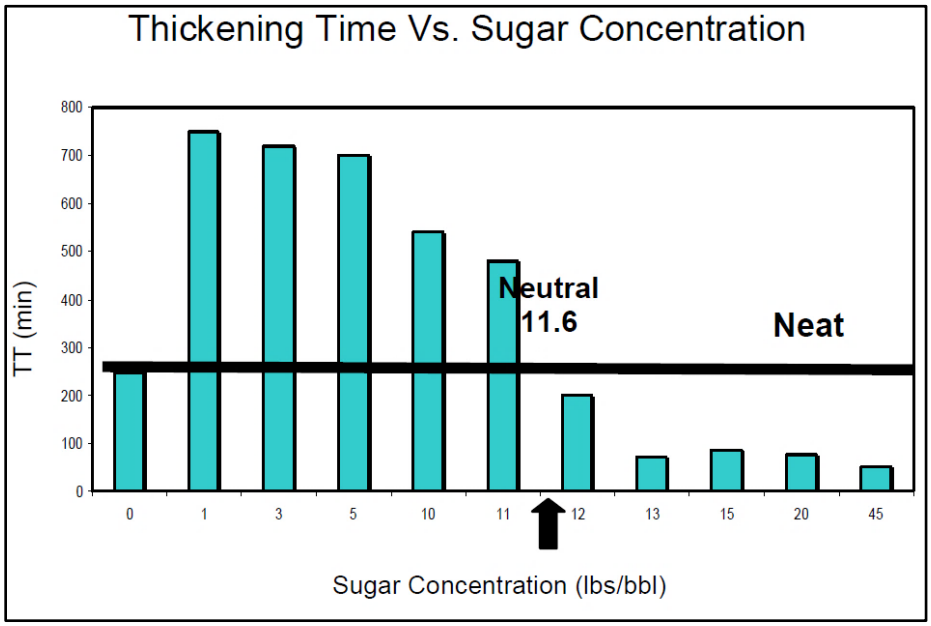

Figure 2.1: Sugar Concentration (Pound/Barrel) Versus Thickening Time of Different Cement Slurries Tested at 26.6 °C [18].

Mazumdar [19] investigated the retardation properties of sugar at different concentrations starting from 0% up to 0.25%. The results revealed that sugar behaves as a retarder at concentrations between 0.01% and 0.08%. However, the use of sugar concentrations in the

range of 0.17%-0.19% leads to an accelerating effect, and sugar acts no longer as a retarder (Figure 2.2).

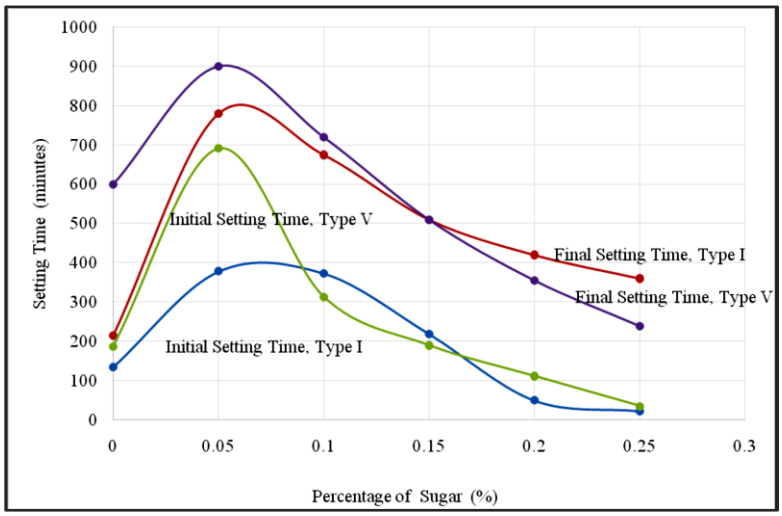

Figure 2.2: Sugar Concentration Versus Setting Time of Cement Paste [19].

Zhang et al. [20] explored the retardation effects of saccharides on the hydration of both, pure C_3S and OPC. The investigated saccharides were also compared with sucrose that is a confirmed retarder. The results indicated that sucrose surpasses the aliphatic sugar alcohols in terms of hydration retardation. Also, the retardation influence of saccharide is more significant in tricalcium silicate than in ordinary Portland cement. They also found that saccharides have an early retardation effect on calcium silicate hydrate precipitation that is obviously greater than it is on the growth of calcium hydroxide in crystalline form. Figure 2.3 presents the relation between the initial setting time of OPC paste and the concentration of the added saccharide.

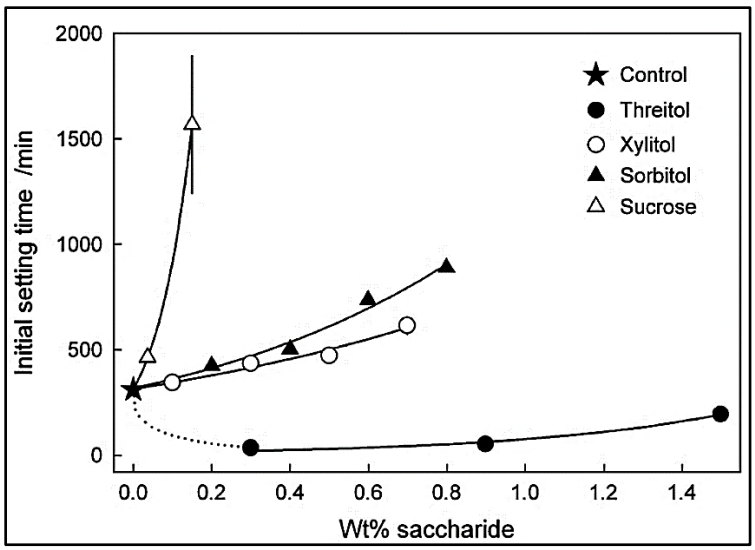

Figure 2.3: Saccharides' Dosage Versus the Initial Setting Time of OPC Paste [20].

Nuruddin et al. [21] considered the use of the glucose sugar as a natural retarder. Its effect of retardation on geopolymer concrete containing fly ash was tested. To represent the actual cast in situ environment, they adopted three curing regimes, namely ambient, external exposure and hot exposure. The results showed that the incorporation of sugar extended the setting time in the mixture. Also, the addition of 1% of sugar had an improvement up to 18.79% relating to the compressive strength in ambient curing environment. Moreover, a 12.75% increment in compressive strength was achieved with the addition of 2% of sugar to samples under external exposure curing method.

Otunyo et al. [22] investigated the possible retardation influence of sugar cane juice on concrete. It was demonstrated that the setting time was extended by partially replacing of water with cane juice. The results also demonstrated that, at all ages, the bulk density increased as the percentage of cane juice increased in the prepared mixtures. Moreover, the strength of the concrete was enlarged and reduced based on the ratio of replacement of sugar cane juice (Figure 2.4).

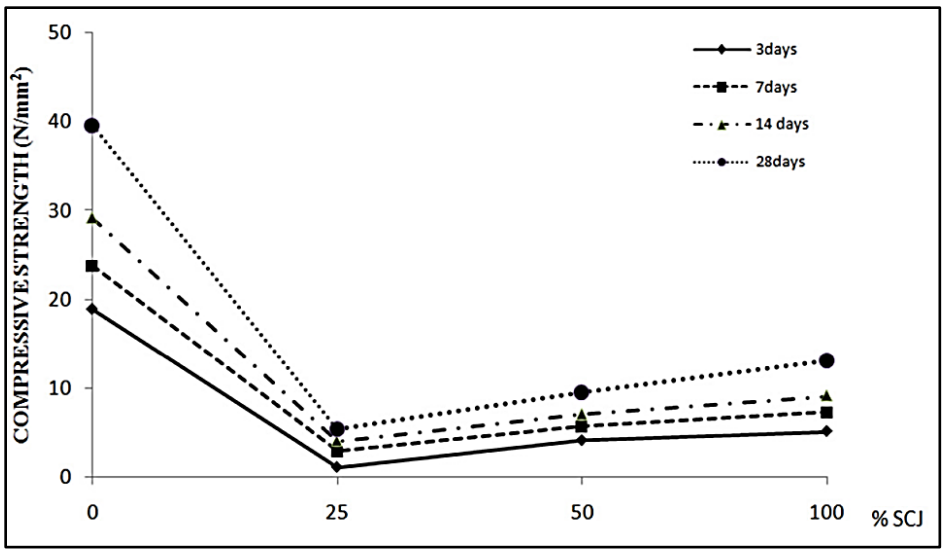

Figure 2.4: Compressive Strength Versus Cane Juice Used as Replacement of Mixing Water [22].

Abalaka [23] studied the retardation influence of sugar on OPC concrete. In that study, sugar was used in concentrations ranging between 0% to 1% of the cement weight. Setting time, soundness and compressive strength were investigated. The results revealed that up to 0.06% sugar, the setting time increased significantly. However, an addition of sugar between 0.2% to 1% cause a flash set. The results also showed that 0.06% sugar enhanced the strength and reduced the expansion of the cement paste A.

2.2.2 Zinc Oxide

Zinc oxide is a common concrete set retarder. Olmo et al. [24] investigated the impact of zinc oxide on cement properties including the time of set and the unconfined compressive strength. The experimental data showed that zinc oxide extended the setting time strongly with reference to that of the cement. However, it decreased the compressive strength at early age, this effect decreased with the sample age.

Zinc oxide is a strong retarding admixture; Berke and Caldarone [25] studied the effects of zinc oxide as a set retarder along with its interaction with supplementary cementitious

materials (SCM's). The used dosages of zinc oxides are: 0.15%, 0.3%, 0.5%, and 1.0%. They suggested that nucleation and or growth poisoning of CSH might be the mechanisms through which zinc oxide retards the hydration reaction. They also found that the SCM's (wheat straw ash, metakaolin, rice straw ash, silica fume and class F fly ash) decrease the retardation time since SCM's offer nucleation sites for CSH precipitation in the cementitious system.

Ataie et al. [26] performed a study to explore the impact of zinc oxide on the hydration process of OPC. Zinc oxide was added between 0.15% to 0.5% by weight of cement. Figure 2.5 presents the evolution of hydration heat with respect to time at varying zinc oxide additions.

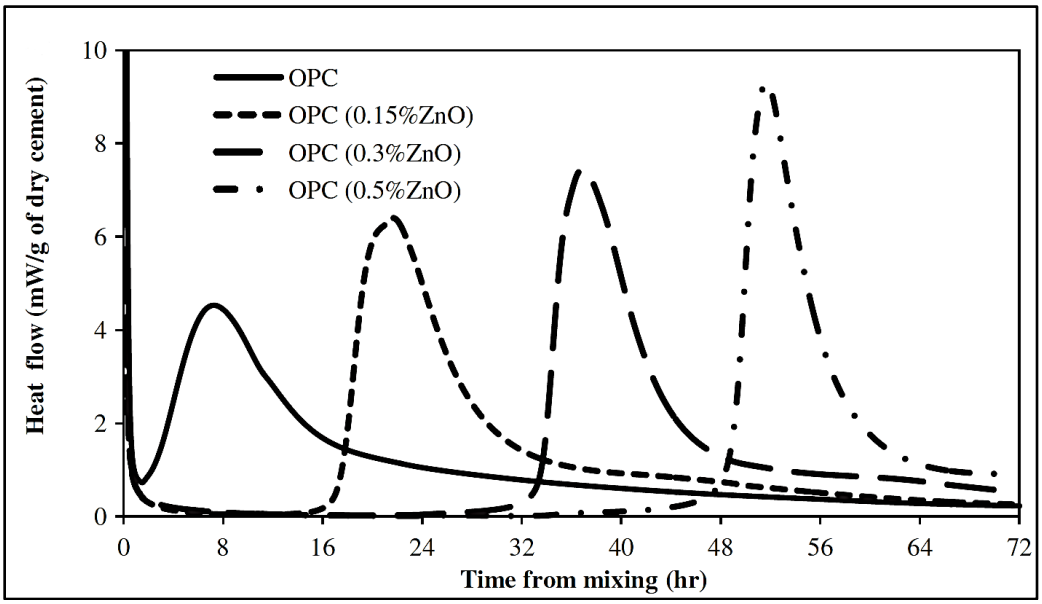

Figure 2.5: Effect of Different ZnO Dosages on the Evolution of Heat of Hydration of OPC [26].

2.2.3 Sodium Gluconate

Sodium gluconate was used to extend the time of set of concrete. Several researchers studied the influence of sodium gluconate as a set retarder. Ma et al. [27] studied the impact

of sodium gluconate on the hydration of cement. They reported that this chemical delays the hydration reaction of tricalcium silicate due to its adsorption that leads to an extended duration of the period of induction. The experimental data revealed that a 1% dosage of sodium gluconate had an important role in inhibiting the reaction of $3CaO \cdot Al_2O_3$ with $CaSO_4 \cdot 2H_2O$. However, a dose of sodium gluconate of less than 0.03% promoted an early-age ettringite formation. The results also revealed that sodium gluconate can be used both, as a strong water reducer and an effective set retarder. Figure 2.6 shows the relation between the dosage of sodium gluconate and the time of set.

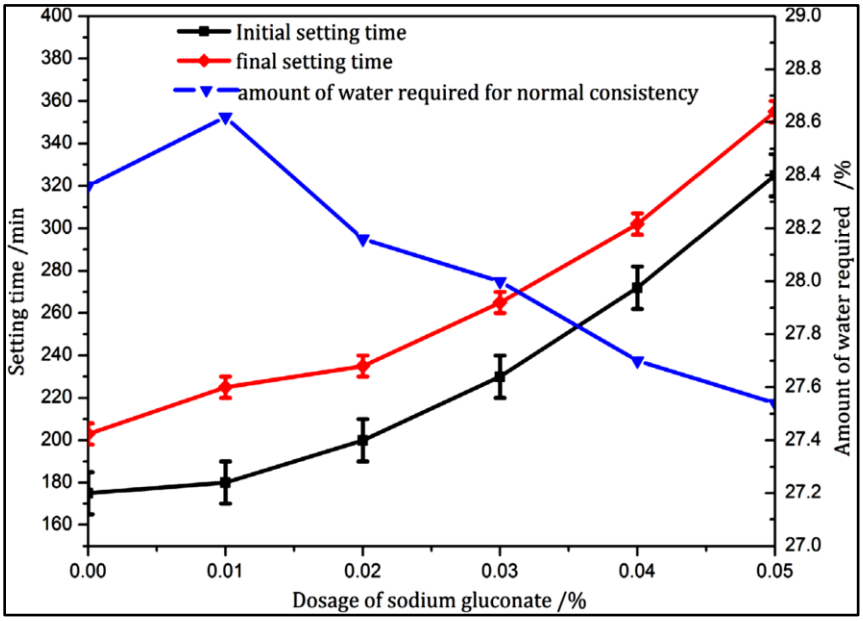

Figure 2.6: Different Sodium Gluconate Dosages Versus Setting Time of Cement Paste [27].

In a previous research by Li et al. [28], the influence of sodium gluconate on the loss of flow and fluidity of cement paste containing superplasticizer of aminosulfonic-acid type was studied (Figure 2.7). The results showed that sodium gluconate resulted in a decrease in the quantity of adsorbed superplasticizer due to the competition between the superplasticizer and the retarder for adsorption over C_4AF–gypsum, C_2S and C_3S surfaces.

The analysis indicated that sodium gluconate retarded the hydration of C_3S and a dosage between 0.03 to 0.09% is recommended for cement pastes to increase fluidity and reduce flow loss.

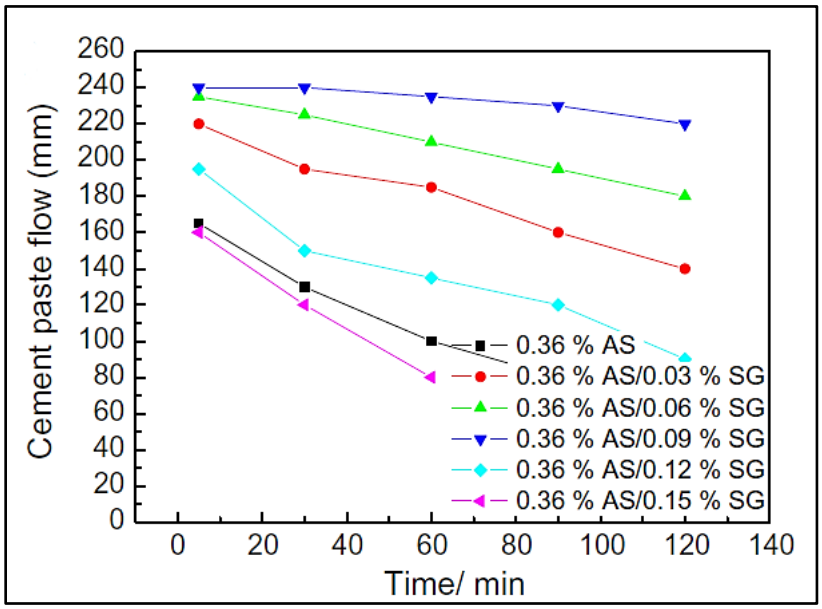

Figure 2.7: Cement Paste Flow Loss at 0.36% Superplasticizer (AS) and Different Sodium Gluconate Dosages [28].

2.2.4 Calcium-Sulfate-Bearing Substances

Some research has been conducted on calcium-sulfate-bearing substances as set retarders. Comparing the retardation characteristics of a mixture of natural gypsum and FGD (flue gas desulfurization) denoted by CGF cement and a mixture of gypsum and anhydrite denoted by CGA cement, in which both containing a clinker content of around 94%, the results show that the FGD gypsum has an improved retardation influence than both the ordinary gypsum and anhydrite (Figure 2.8), even though they both included $CaSO_4.2H_2O$ in their composition. Comparing the compressive strength of both cements, the anhydrite improved the 28-day compressive strength which is significantly apparent at 20% anhydrite content of the total added calcium-sulfate-bearing substance (Figure 2.9) [29].

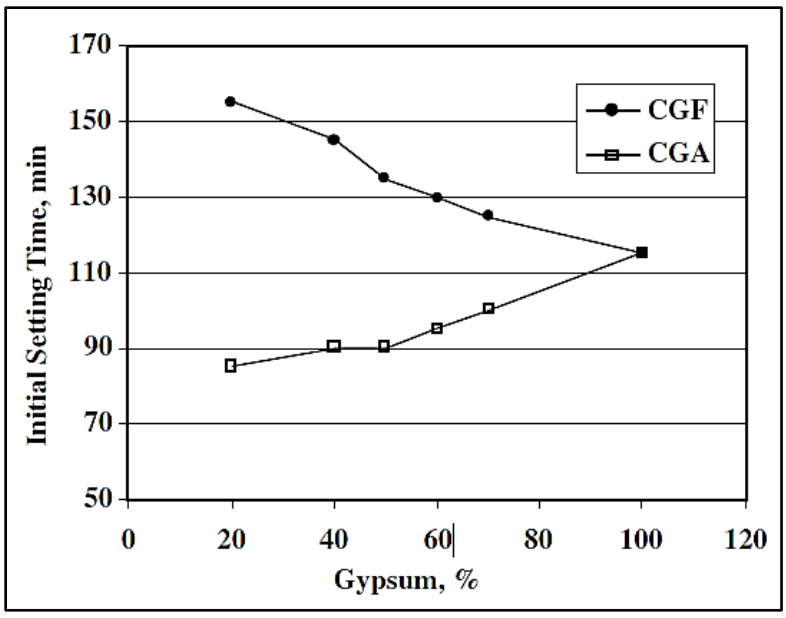

Figure 2.8: Initial Setting Time of CGF and CGA cements at different Gypsum Contents [29].

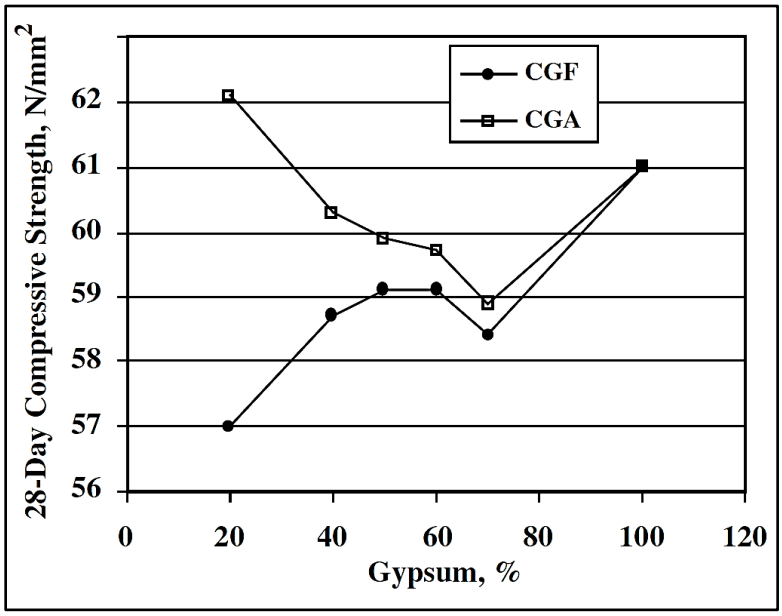

Figure 2.9: 28-Day Compressive Strength of CGF and CGA cements at different Gypsum Contents [29].

Tzouvalas et al. [30] considered the replacement of natural gypsum by natural anhydrite as a set retarder. The experimental data indicated that anhydrite could be an alternative to natural gypsum depending on strength and setting time results. They also reported that a mixture of anhydrite and gypsum appears to be more efficient in controlling the setting

time. In another work by Tzouvalas et al. [31], the authors investigated the replacement of natural gypsum by FGD gypsum as a set retarder. FGD gypsum is an industrial byproduct of a process called flue gas desulfurization. Both gypsums were used in mixes up to 7.5% of the cement weight. The experimental data revealed that FGD gypsum could be a substitute to natural gypsum depending on strength and setting time results. The results also revealed that a blend of FGD gypsum and natural gypsum appears to be more efficient in controlling the setting time.

Chandara et al. [32] replaced the natural gypsum by waste gypsum in the manufacture of ordinary Portland cement. Waste gypsum was recycled from slip casting molds in the ceramic industry. Both gypsums were used according to the following proportions: 0%, 3%, 4% and 5%. The results showed that natural gypsum exhibited a longer setting time compared to the waste gypsum. However, the mechanical properties were not sacrificed in both cases.

Medina et al. [33] tried to recycle calcium sulfate from waste plasterboards and drywalls. The results showed that the treated recycled calcium sulfate could be used to prevent flash setting, so it can be considered a set retarder. However, the origin of the recycled calcium sulfate needs to be undertaken to retain the strengths when hardened.

Shen et al. [34] carried out a study on the replacement of natural gypsum by anhydrite in circulating fluidized bed combustion ashes as a set retarder. Volume stability, strength development, setting time and hydration process have been observed. The experimental data showed that the anhydrite in circulating fluidized bed combustion ashes is an appropriate replacement to gypsum to perform as an effective setting retarder. However, it

reduced the mortar strength in comparison to natural gypsum. The research reported that the anhydrite in circulating fluidized bed combustion ashes makes a slower and more transformable ettringite formation compared to natural gypsum and it can be effectively used as a cementing constituent with proper proportions design.

Ozkul [35] performed a study to explore the retardation behavior of citrogypsum and desulphogypsum. Setting time, volume stability and other properties of cement pastes prepared by adding both chemical gypsums were reported. The results demonstrated that the setting time was within the standard limits. However, pastes made with citrogypsum showed some decrease in the initial time of set while pastes made with desulphogypsum exhibited a one-hour delay in terms of final time of set compared to natural gypsum.

Altun and Sert [36] replaced natural gypsum by phosphogypsum in cement. Both gypsums were used according to the following proportions: 1%, 3%, 5%, 7%, 10%, and 12.5%. Setting time, volume expansion and compressive strength were measured. The results showed that phosphogypsum is a good cement set retarder. The results also showed that 3% phosphogypsum recorded the highest 28-day compressive strength.

Shen et al. [37] also carried out a study on calcium-sulfate-bearing substances as set retarders. The research compared the retardation effects of solidified phosphogypsum and natural gypsum with and without fly ash supplementary material. The solidified phosphogypsum was used up to 10% cement replacement. Both types of gypsums exhibited similar setting behavior. However, solidified phosphogypsum produced a higher strength cement.

2.2.5 Other Substances

Bishop and Barron [38] investigated the inhibition of cement hydration with lignosulfonate, tartaric acid and sucrose. The results showed that lignosulfonate and sucrose accelerate the formation of ettringite. However, they are more efficient at the retardation of C_3S hydration. The results also showed that the most efficient retarder for the hydration of C_3A and the formation of ettringite is tartaric acid.

Singh [39] studied the hydration of Portland cement including 10% rice husk ash in the presence of 1% lignosulfonate. The results showed that 1% lignosulfonate significantly enhanced the setting time of Portland cement (Figure 2.10). However, 1% lignosulfonate reduced the compressive strength slightly after hydration for 7 and 28 days.

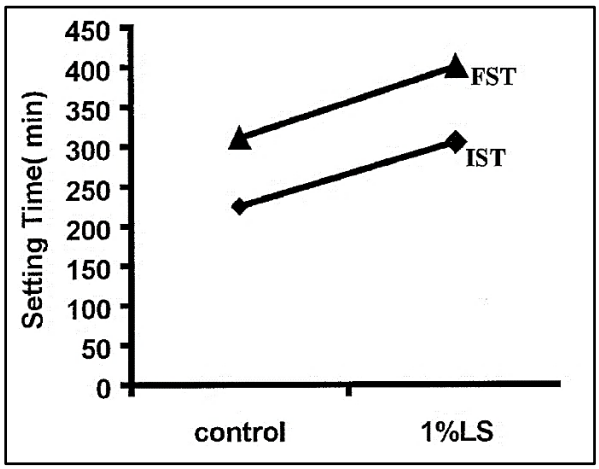

Figure 2.10: Impact of 1% Lignosulfonate on the Setting Time of Portland Cement [39].

The retardation effect of tartaric acid was also studied by Rai et al. [40]. They showed that tartaric acid has a strong retardation impact on the hydration of OPC. Tartaric acid retards the hydration through the generation of a layer on cement grains' surface. Therefore, it blocks the hydration. Figure 2.11 shows the heat evolution rate at 0% and 1% tartaric acid addition to OPC.

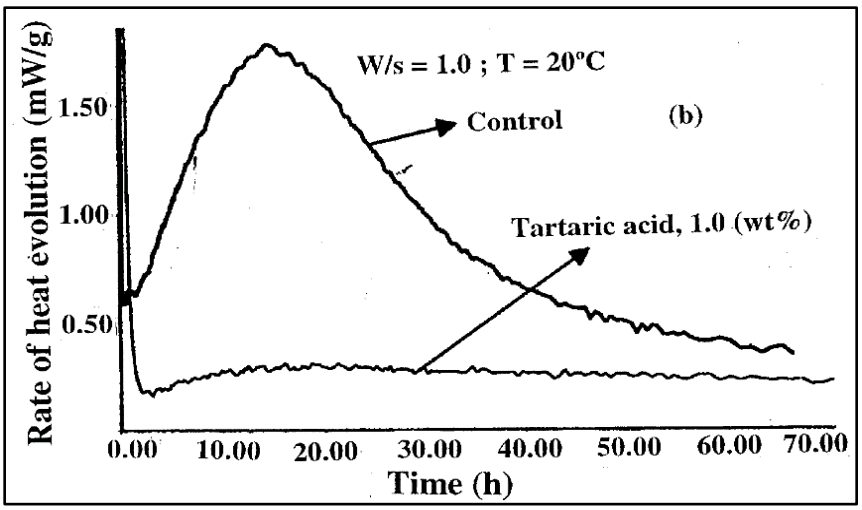

Figure 2.11: Rate of Heat Evolution at 0% and 1%Tartaric Acid Addition to OPC [40].

2.3 EFFECT OF INDUSTRIAL BYPRODUCTS ON CEMENT RETARDATION

2.3.1 Electric Arc Furnace Dust

Electric arc furnace dust (EAFD) is a byproduct that originates from steel making industry. Figure 2.12 elaborates the process which results in the formation of EAFD as a byproduct of steel making [41]. Al-Zaid et al. [42] carried out a study on the use of EAFD in the manufacture of concrete. They investigated the influence of EAFD on properties of fresh and hardened concrete, this includes the setting time, compressive strength, drying shrinkage and others. The experimental data revealed that EAFD retards the setting time steadily; the more the byproduct used, the longer the resulted setting time. Moreover, the engineering properties seem to be improved with the use of EAFD. The experimental program used up to 5% EAFD to study the setting time. Later, an emphasis was given to 2%-3% of EAFD.

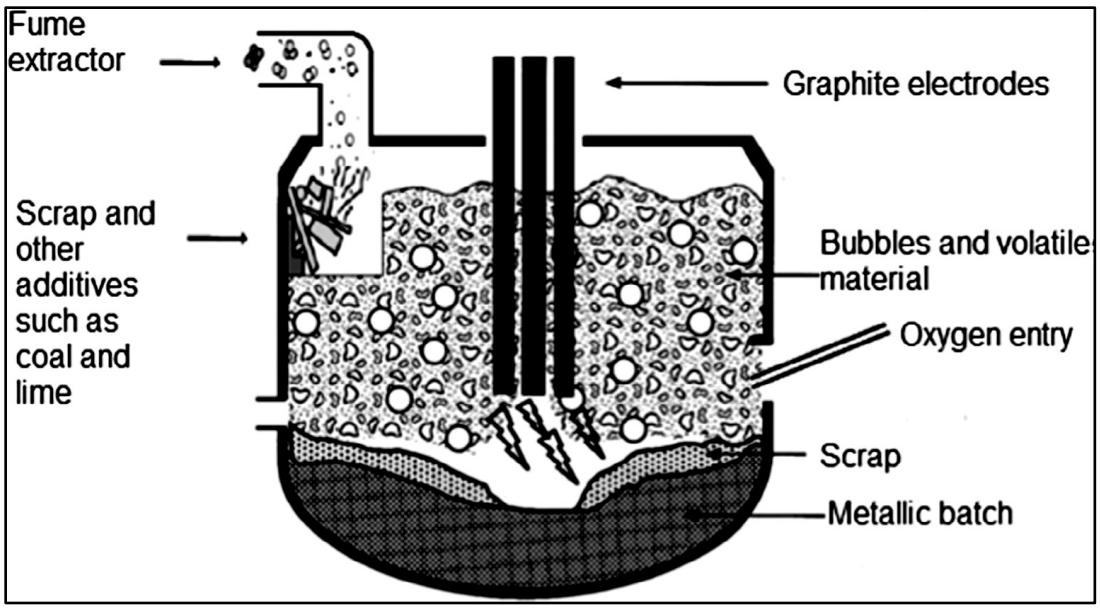

Figure 2.12: Formation of EAFD in steelmaking Industry [41].

Maslehuddin et al. [43] conducted a study to assess the mechanical and durability properties of concrete made from OPC and blended cement (fly ash and silica fume) prepared with 0% and 2% EAFD. The quantities of cementitious materials used to prepare concrete is briefly summarized in Table 2.1. Addition of 2% EAFD extended the setting time of concrete prepared in the study. The research also showed an improvement to the compressive strength using 2% EAFD in concrete (Figure 2.13). Moreover, 2% EAFD improved the workability and slump retention (Figure 2.14).

Table 2.1: Materials Combinations Used in the Study [43].

Mix	OPC (%)	EAFD (%)	SF (%)	FA (%)
Typ1	100	0	0	0
Typ1 + EAFD	98	2	0	0
Typ1 + SF	92	0	8	0
Typ1 + SF + EAFD	92	2	6	0
Typ1 + FA	70	0	0	30
Typ1 + FA + EAFD	70	2	0	28

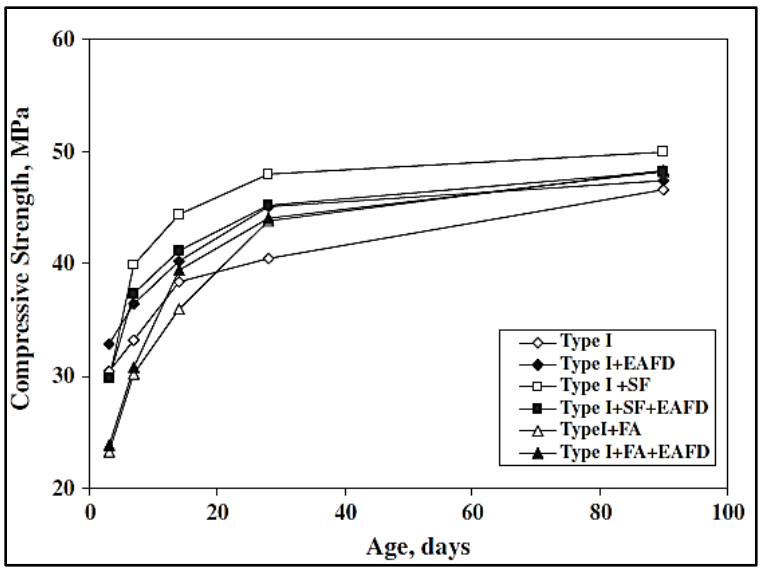

Figure 2.13: Compressive Strength Versus Age for OPC and Blended Cements Concretes [43].

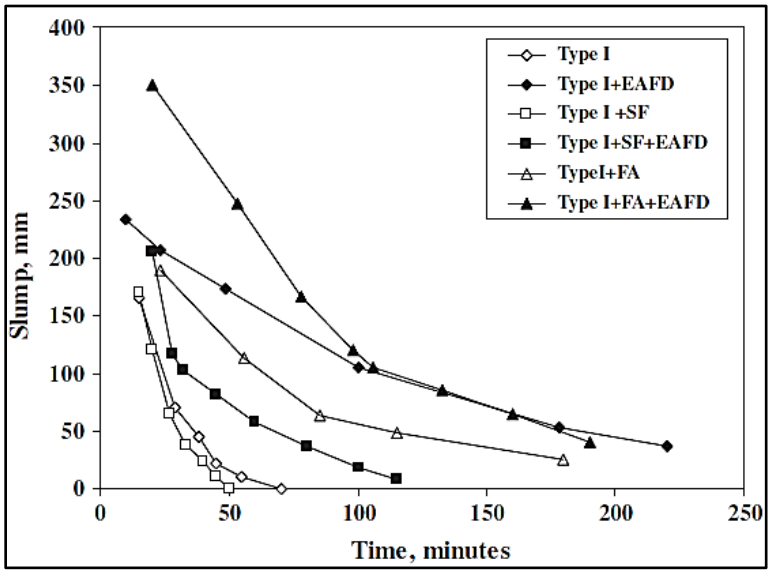

Figure 2.14: Slump Retention Versus Time for OPC and Blended Cements Concretes [43].

Fares et al. [44] investigated the use of electric arc furnace dust as a fine powder to produce green concrete. In their study, they tested cement with different quantities of fresh EAFD to obtain the optimum dosage. The effect of EAFD was compared to mortars containing fly ash and silica fume, according to several water to binder ratios and different sand to cement ratios. The results showed an alternation of the reaction between zinc and calcium in C_3A that produced a considerable disappearance in ettringite crystals. It was concluded

that EAFD has a high content of zinc and heavy metals that react with the different cement phases leading to a high retardation of the hydration process. Thus, an extended setting time. Figure 2.15 and Figure 2.16 show the effect of 0% and 3% EAFD on the setting time and the hydration temperature, respectively.

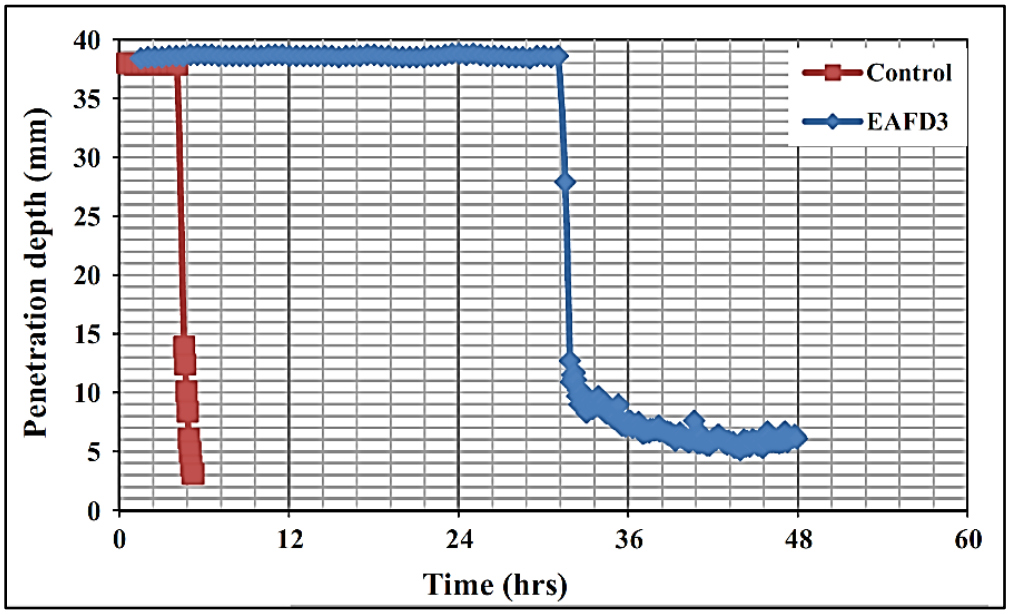

Figure 2.15: Penetration Depth Versus Time for Setting Time Evaluation [44].

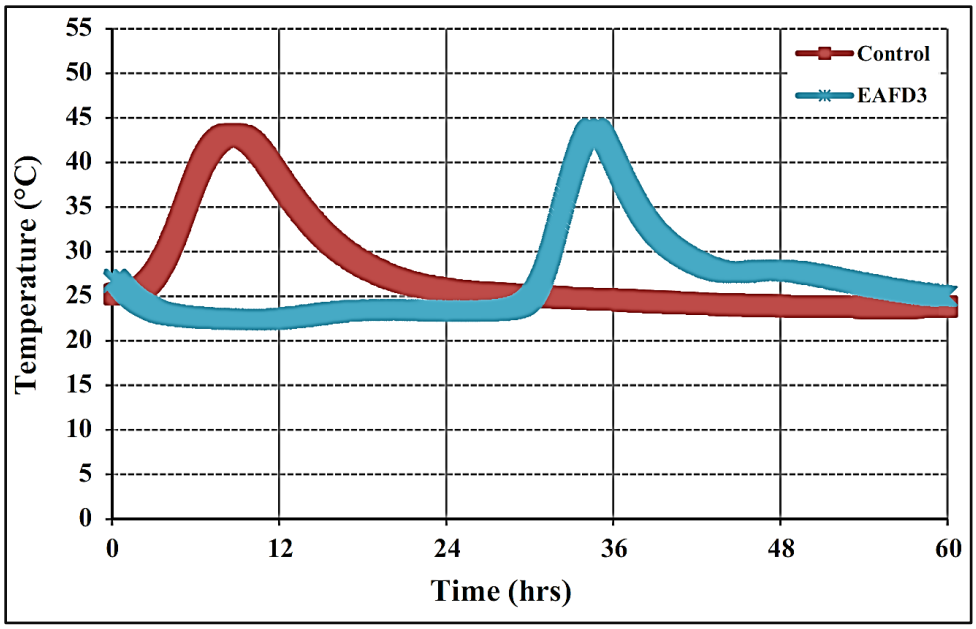

Figure 2.16: Evolution of Temperature of Hydration with Time [44].

2.3.2 Oil Ash

Little information in the literature is available describing the effect of oil ash on the setting time of Portland cement. Camilleri et al. [45] studied the effect of oil ash, resulted from the combustion of oil, as a replacement in mortar and concrete. It was reported that oil ash caused and increase in the setting time of tested mortars. However, the use of oil ash reduced the compressive strength in the tested mortar and in the hardened flowable concrete.

2.3.3 Limestone Powder

The retardation effect of LSP is debatable in the literature. Bentz et al. [46] tried to determine the effects of adding LSP to control the setting of mixtures containing a high percentage of fly ash. The results showed an accelerating effect due to using 5% LSP which reduced the initial and final setting time. Similarly, Mohammadi and South [47] used a higher limestone content (4.5-12.5%) with general purpose and blended cements (silica fume and fly ash). High strength concrete specimens were prepared. The results indicated that LSP has a minor influence on the improvement of workability. Furthermore, field concrete mixes exhibited a decrease in setting time that was more obvious in the initial setting time.

On the other hand, Felekoglu [48] incorporated the quarry waste LSP at 10% substitution. The results showed that LSP reduced the superplasticizer requirements in self-compacted concrete. In a study conducted by Heikal et al. [49], it was revealed that a 5% replacement of cement by limestone dust increased the final setting time of fly ash-limestone-filled pozzolan cement. Dhir et al. [50] stated that the addition of LSP in cement extends the

initial setting time. Similarly, Tosun et al. [51] highlighted that the incorporation of LSP in cement prolonged the initial setting time. Furthermore, Gesoğllu et al. [52] noted that substituting cement with LSP caused an increase in the setting time.

2.4 SET RETARDERS IN HOT WEATHER CONDITIONS

Several researchers tried to study the effects of set retarders in hot weather conditions based on setting time and other mechanical properties. Khan and Ullah [53] examined the impact of "ASTM C 494 Type D" retarding admixture on setting time of cement under three curing regimes with different temperatures and relative humidity. Three types of cement were considered to make the tested pastes. The authors concluded that the admixture affects the setting time depending on the admixture dosage, the cement type and the cutting regime. Generally, the admixture extended the setting time. However, in one cement type, exceeding a dosage of 0.25% reduced the initial setting time and extended the final setting time. The results also showed that higher temperatures and lower humidity accelerate the setting time in all pastes regardless of the admixture use. Table 2.2 provides the setting time (in minutes) of Type III cement for varying retarder dosages at three curing regimes. Table 2.3 represents the data provided in Table 2.2 relative to the case of 0% retarder at the first curing regime.

Table 2.2: Setting Time (min.) of Type III Cement for Varying Retarder Dosages at Three Curing Regimes [53].

Retarder (%)	22 °C, 55-65% RH		35 °C, 35-45% RH		50 °C, 25-35% RH	
	IST	FST	IST	FST	IST	FST
0	113	202	96	138	76	117
0.125	260	355	188	260	112	205
0.250	456	700	300	405	125	296
0.375	≅600	≅708	353	638	128	329

Table 2.3: Retardation due to the Retarder Relative to the Case of 0% Retarder at the First Curing Regime [53].

Retarder (%)	22 °C, 55-65% RH		35 °C, 35-45% RH		50 °C, 25-35% RH	
	IST	FST	IST	FST	IST	FST
0	1.000	1.000	0.850	0.683	0.673	0.579
0.125	2.300	1.757	1.664	1.287	0.991	1.105
0.250	4.040	3.465	2.655	2.005	1.106	1.465
0.375	5.310	3.505	3.124	3.158	1.133	1.629

Otoko [10] investigated the use of cassava powder, which is an industrial byproduct, as an admixture in hot weather concreting practices. Concrete cube specimens were prepared and submerged at ambient, 40 °C and 60 °C curing temperatures. The influence of the admixture on setting time and compressive strength was monitored. The strength of cubes was tested at variable days of maintained curing. The experimental data showed an ability of cassava powder to increase the workability and retard the setting time of concrete. It also increased the early and late strength of concrete.

A considerable amount of research was devoted to investigate the effects of different materials as concrete set retarders. Previous research assessed the retardation effects considering mechanical and durability properties of produced concrete. The research showed different rates of retardation corresponding to the different amounts of the materials used. Also, the mechanical and durability properties varied in various manners to the added admixtures.

Most existing commercial retarding admixtures are costly and have a short service life. This heightens the need for set retarders which are more economical and have a long shelf life. This literature review provided important insights into the methods and tests adopted in earlier studies for the quantitative verification of the influence of set retarders on fresh and hardened concrete. Available experimental data presented in the literature seems to

provide evidence that the selected indigenous industrial byproducts have promising retarding effects. However, there are several properties, other than setting time, that need to be verified to assure that these industrial byproducts are fit to be added to concrete. They should not adversely affect the properties of concrete, such as compressive strength and drying shrinkage, to meet the demands of standards, industry and market.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter is devoted to the discussion and the explanation of the experimental work carried out in this study. It also describes the materials needed and mixture proportions. Besides, a brief review of the tests performed is presented.

3.2 MATERIALS

3.2.1 Cement

Type I cement satisfying the requirements of ASTM C150 cement was used in the preparation of all the concrete mixtures. It has a specific gravity of 3.15. Table 3.1 and Table 3.2 show the oxide and compound composition of the cement used in this study, respectively. Figure 3.1 shows a picture of the used ordinary Portland cement.

Table 3.1: Oxide Composition of Type I Cement

Component	Wt.%
CaO	64.35
SiO_2	22.00
Al_2O_3	5.64
$Fe_2O_{3)}$	3.80
K_2O	0.36
Na_2O	0.19
MgO	2.11
Equivalent Alkalis	0.33
SO_3	2.10
LOI	0.70

Table 3.2: Major Compound Composition of Type I Cement.

Component	Wt.%
C_3S	55
C_2S	19
C_3A	10
C_4AF	7

Figure 3.1: Ordinary Portland Cement.

3.2.2 Aggregate

i. Fine Aggregate

Locally sourced dune sand brought from Eastern Province in Saudi Arabia with a specific gravity of 2.56 and an approximate water absorption of 0.5% was used as fine aggregate. Figure 3.2 shows the used fine aggregate that is mostly quartz.

Figure 3.2: Fine Aggregate.

ii. Coarse Aggregate

Table 3.3 gives four sizes of crushed coarse aggregate used in the concrete mixtures in this study. Coarse aggregate was obtained from a local limestone quarry in Saudi Arabia. The specific gravity of the coarse aggregate was around 2.6 and its water absorption was approximately 1.1%. Figure 3.3 shows the used coarse aggregate.

Table 3.3: Coarse Aggregates Grading.

Size of Sieve	Retained (%)	Cumulative Retained (%)	Passing (%)
3/4 in	0	0	100
1/2 in	30	30	70
3/8 in	30	60	40
3/16 in	35	95	5
3/32 in	5	100	0

Figure 3.3: Coarse Aggregate.

3.2.3 Retarders

i. Conplast RP264®

This retarder was procured from a local supplier in Saudi Arabia. Table 3.4 summarizes some technical information of the retarder provided by the manufacturer. Figure 3.4 shows Conplast RP264® in a measuring beaker.

Table 3.4: Technical Information of Conplast RP264®.

Property	Description
Type	Lignosulphonate
Specific Gravity @ 20ºC	1.16 + 1.5%
Chloride Content (BS 5075)	Nil
Alkali Content (Na_2O)/ Liter	Less than 5.0 g

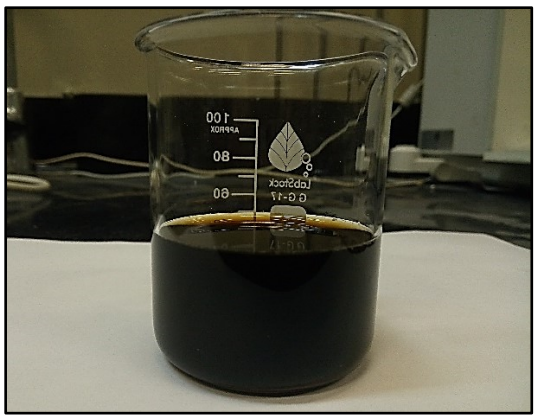

Figure 3.4: Conplast RP264® Retarder.

ii. Sika Retarder 21®

This retarder was procured from a local supplier in Saudi Arabia. Table 3.5 shows some technical information of the retarder as supplied by the manufacturer. Figure 3.5 shows Sika Retarder®21 in a measuring beaker.

Table 3.5: Technical Information of Sika Retarder 21®.

Property	Description
Type	Based on Phosphates
Density @ 25°C	1.09 kg/L
Chloride Content (EN 934-2)	Nil

Figure 3.5: Sika Retarder 21®.

iii. Sugar

It is the common table sugar which is composed entirely of sucrose ($C_{12}H_{22}O_{11}$) (Figure 3.6).

Figure 3.6: Sugar.

iv. Sodium Gluconate

Sodium gluconate (sodium salt of gluconic acid) was procured from a local supplier in Saudi Arabia. It has the chemical formula $C_6H_{11}NaO_7$. Figure 3.7 shows a picture of sodium gluconate.

Figure 3.7: Sodium Gluconate.

v. Zinc Oxide

Zinc oxide (ZnO) was procured from a local supplier in Saudi Arabia. Figure 3.8 shows a picture of zinc oxide.

Figure 3.8: Zinc Oxide.

3.2.4 Superplasticizer

Conplast SP430® was procured from a local supplier in Saudi Arabia. It was used in dosages between 0.6% to 0.8% by cement weight. Table 3.6 shows some technical information of the superplasticizer as provided by the manufacturer. Figure 3.9 shows Conplast SP430® in a measuring beaker.

Table 3.6: Conplast SP430®.

Property	Description
Type	Sulphonated Naphthalene Polymers
Specific Gravity @ 20ºC	1.2
Chloride Content (BS 5075)	Nil
Alkali Content (Na_2O)/ Liter	Less than 72.0 g

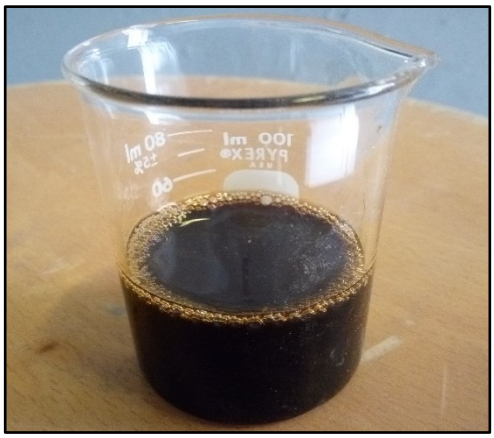

Figure 3.9: Conplast SP430® Superplasticizer.

3.2.5 Indigenous Industrial Byproducts

i. Electric arc furnace dust (EAFD)

This byproduct was brought from a local steelmaker in Saudi Arabia. Table 3.7 describes the major constituents of EAFD. The material was used in both the untreated and the treated (T-EAFD) states. Thermal treatment was done at 800ºC for 2 hours. Figure 3.10 and 3.11 show pictures of untreated and treated EAFD, respectively.

ii. Oil Ash

This byproduct was obtained from a local power plant in Saudi Arabia. Table 3.8 describes the major constituents of oil ash. Figure 3.12 shows a picture of oil ash.

Table 3.7: Major Constituents of Electric Arc Furnace Dust (EAFD).

Constituent	Weight (%)
Iron	33.6
Zinc	10
Calcium	9.39
Sodium	2.6
Silicon	2.38
Magnesium	2.3
Manganese	1.8
Potassium	1.7
Lead	1.31
Aluminum	0.7
Sulphur	0.57
Phosphorous	0.13
Titanium	0.09
Copper	0.06
Tin	0.03
Nickel	0.01
Cadmium	0.0004

Table 3.8: Major Constituents of Oil Ash.

Constituent	Weight (%)
Sulfur	9.6
Vanadium Oxide	2.65
Silica	1.65
Ferric Oxide	0.47
Sodium Oxide	0.53
Magnesium Oxide	0.48
Equivalent Alkalis	0.55
Calcium Oxide	0.45
Aluminum Oxide	<Less than 0.10
Potassium Oxide	0.03
Loss on Ignition	60.6
Moisture	5.9

Figure 3.10: EAFD Without Heat Treatment.

Figure 3.11: Thermally-Treated EAFD.

Figure 3.12: Oil Ash.

3.2.6 Dosages of the Materials Used in Setting Time Trials

Several trials were conducted to evaluate of the effect of the selected set retarders on the initial and final setting time. Tables 3.9 through 3.11 present the dosages of each material used in the setting time trials.

Table 3.9: Dosages of Commercial Retarders.

Retarder	Dosage
Conplast RP264®	0.35%
	0.40%
	0.65%
	0.95%
Sika Retarder 21®	0.1%
	0.2%
	0.8%
	1.2%
	1.5%

Table 3.10: Dosages of Industrial Byproducts.

Material	Dosage
EAFD	0.5%
	1%
	2%
	3%
	4%
	5%
	6%
T-EAFD	5%
	10%
	30%
	40%
Oil Ash	0.5%
	1.0%
	1.5%
	2%
	3%

Table 3.11: Dosages of Chemicals.

Material	Dosage
Sugar	0.01%
	0.02%
	0.03%
	0.04%
	0.05%
	0.10%
	0.18%
	0.25%
	0.80%
ZnO	0.02%
	0.05%
	0.08%
	0.09%
	0.15%
	0.50%
	1.00%
Sodium gluconate	0.01%
	0.02%
	0.05%
	0.06%
	0.08%

3.2.7 Mortar and Concrete Mix Proportions

To accomplish the other objectives of this study, two types of mixes were prepared after the selection of the retarder dosage depending on the setting time results.

i. Mortar Mixes

Mortar specimens were prepared to study effects of the selected set retarders on the drying shrinkage. Table 3.12 shows details of mortar mixes used in this work.

Table 3.12: Details of Mortar Mixes Components.

Material	Dosage (%)	Weight (g)	Cement (g)	w/c Ratio	Fine Aggregate (g)
OPC	--	--	750	0.4	1500
EAFD	0.5	3.75	750	0.4	1496.3
	1	7.5	750	0.4	1492.5
	2	15	750	0.4	1485
	3	22.5	750	0.4	1477.5
T-EAFD	30	225	750	0.4	1275
	40	300	750	0.4	1200
Oil Ash	1.5	11.25	750	0.4	1488.8
	2	15	750	0.4	1485
	3	22.5	750	0.4	1477.5
Conplast RP264®	0.35	2.61	750	0.4	1500
Sika Retarder 21®	0.8	6	750	0.4	1500
Sugar	0.03	0.225	750	0.4	1499.8
ZnO	0.08	0.6	750	0.4	1499.4
Sodium Gluconate	0.05	0.375	750	0.4	1499.6

ii. Concrete Mixes

Based on setting time trials, several concrete mixes were prepared to investigate the influence of the selected set retarders on the mechanical properties of concrete. Table 3.13 provides details of concrete mixes used to determine the compressive strength. Later, the

mixes were slightly modified in order to increase the initial slump to be suitable for performing the slump retention test, as shown in Table 3.14.

Table 3.13: Details of Concrete Mixes Components.

Material	Material Addition (%)	Material Weight (kg/m^3)	Cement (kg/m^3)	Water (kg/m^3)	CA (kg/m^3)				FA (kg/m^3)
					1/2 in.	3/8. in.	3/16 in.	3/32 in.	
OPC	--	--	375	166.9	339.9	339.9	396.6	56.7	755.4
EAFD	0.5	1.875	375	166.9	339.6	339.6	396.2	56.6	754.8
	1	3.75	375	166.9	339.3	339.3	395.9	56.6	754.0
	2	7.5	375	166.9	338.7	338.7	395.1	56.4	752.6
	3	11.25	375	166.9	338.1	338.1	394.4	56.3	751.2
T-EAFD	30	112.5	375	166	321.0	321.0	374.5	53.5	713.3
	40	150	375	165.7	314.7	314.7	367.1	52.4	699.3
Oil Ash	1.5	5.63	375	166.8	335.6	335.6	391.5	55.9	745.8
	2	7.50	375	166.7	334.2	334.2	389.8	55.7	742.6
	3	11.25	375	166.5	331.2	331.2	386.4	55.2	736.1
Conplast RP264®	0.35	1.305	375	166.9	339.4	339.4	396.0	56.6	754.3
Sika Retarder 21®	0.8	3	375	166.9	338.7	338.7	395.1	56.4	752.6
Sugar	0.03	0.113	375	166.9	339.9	339.9	396.6	56.7	755.4
ZnO	0.08	0.300	375	166.9	339.9	339.9	396.6	56.7	755.4
Sodium Gluconate	0.05	0.188	375	166.9	339.8	339.8	396.5	56.6	755.2

Table 3.14: Details of Concrete Mixes Components for Slump Test.

Material	Material Addition (%)	Material Weight (kg/m^3)	Cement (kg/m^3)	Water (kg/m^3)	CA (kg/m3)				FA (kg/m^3)
					1/2 in.	3/8 in.	3/16 in.	3/32 in.	
OPC	--	--	375	187.51	330.2	330.2	385.2	55.0	733.8
EAFD	0.5	1.875	375	187.49	329.9	329.9	384.9	55.0	733.1
	1	3.75	375	187.48	329.6	329.6	384.5	54.9	732.4
	2	7.5	375	187.45	328.9	328.9	383.8	54.8	731.0
	3	11.25	375	187.42	328.3	328.3	383.0	54.7	729.6
T-EAFD	30	112.5	375	193.52	307.9	307.9	359.2	51.3	684.3
	40	150	375	195.04	300.7	300.7	350.9	50.1	668.3
Oil Ash	1.5	5.63	375	187.29	325.8	325.8	380.1	54.3	724.1
	2	7.50	375	187.22	324.4	324.4	378.4	54.1	720.8
	3	11.25	375	187.07	321.5	321.5	375.1	53.6	714.4
Conplast RP264®	0.35	1.312	375	187.48	329.7	329.7	384.6	54.9	732.6
Sika Retarder 21®	0.8	3	375	187.45	328.9	328.9	383.7	54.8	730.9
Sugar	0.03	0.113	375	187.51	330.2	330.2	385.2	55.0	733.7
ZnO	0.08	0.300	375	187.51	330.2	330.2	385.2	55.0	733.7
Sodium Gluconate	0.05	0.188	375	187.50	330.1	330.1	385.1	55.0	733.5

3.3 TESTS

Details of tests conducted on paste, mortar and concrete specimens are shown in Tables 3.15 and 3.16 in which test method, specimen type, specimen dimensions and test age are presented for each studied property.

Table 3.15: Fresh Properties.

Property	Method	Specimen Type
Setting time	ASTM C191	Paste
Heat of hydration	Calorimetry	Paste
Slump Retention	ASTM C143	Concrete

Table 3.16: Hardened Properties.

Property	Method	Specimen Type	Specimen Shape and Size	Test Age
Compressive strength	ASTM C39	Concrete	Cylinder: 75x150 mm	3, 7, 14, 28 and 90 days of water curing
Drying shrinkage	ASTM C596 ASTM C157	Mortar	Prism: 25x25x285 mm	4, 7, 14, 28, 56 and 90 days starting after 28 days of water curing
Morphology	SEM	Paste	Cube: 50x50x50 mm	28 days of water curing

3.3.1 Setting Time

The experimental work was started by investigating the influence of the addition of each material on the initial and final setting time. The conducted setting time tests are the trials that were employed for the selection of mortar and concrete mixes. Promising dosages that gave acceptable extended setting time were selected to conduct the other tests.

Setting is a term that usually defines the state at which cement paste starts to stiffen. After the initial set, the cement paste is significantly stiff, so it cannot be molded anymore. Final

setting means that the paste is hard enough to sustain a slight load. In this study, Vicat needle apparatus was used to determine the setting time according to the procedure described in ASTM C191 [54]. Setting time was determined from the instant of adding water. According to the mentioned standard, the initial setting time is the time needed for the 1-mm Vicat needle to penetrate 25 mm into the cement paste. The final setting time is determined at the instant just when the 1-mm needle does not sink visibly into the paste. Figure 3.13 depicts the apparatus used to determine the setting time. The various parts of the Vicat's apparatus are illustrated diagrammatically in Figure 3.14. It is composed of a main frame (A), a rod capable of being moved (B), a 10-mm diameter end (C), Vicat's 1-mm diameter needle (D), a screw (E) which is used to secure and release the rod B, an indicator (F), Vicat's mold (G) and a nonabsorbent plate (H) [55]. Some specimens prepared for the setting time test are shown in Figure 3.15. As stated in ASTM C150 [56], the minimum initial setting time is 45 minutes. Limits on the final setting time appear no more in the ASTM or European standards [2]. However, in this study, a maximum time of 500 minutes was chosen as the final setting time.

Figure 3.13: Vicat Needle Apparatus.

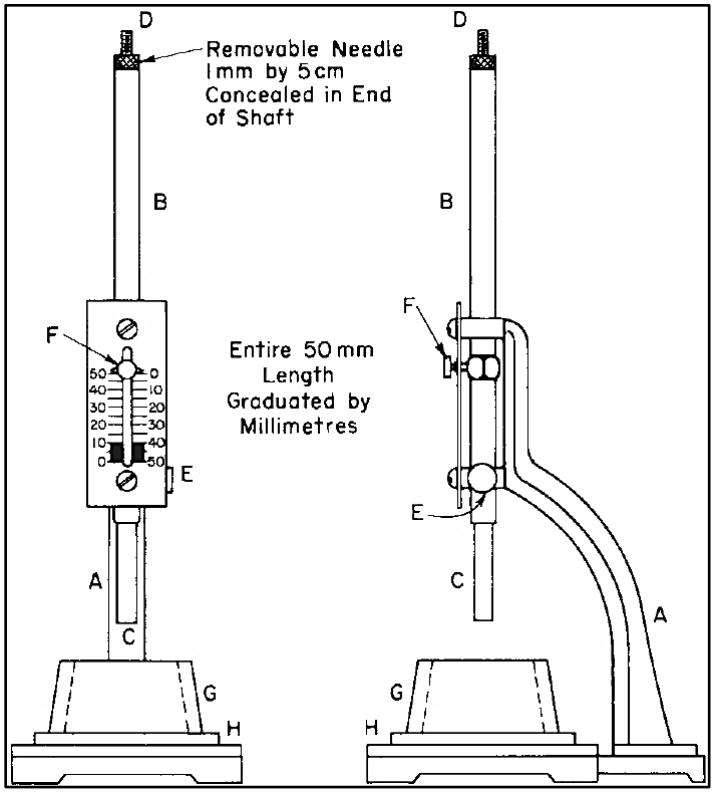

Figure 3.14: Parts of Vicat Needle Apparatus [55].

Figure 3.15: Specimens Molded to Determine the Setting Time.

3.3.2 Heat of Hydration

Hydration of cement is an exothermic reaction [2]. Higher thermal stresses may develop due to casting of large quantities of concrete in structures with large volumes such as dams and foundations having great thicknesses. Thus, it is crucial to reduce the rate of heat generation by using the appropriate cement type, additives and/or any other precautionary measures [57].

In this study, the evolution of heat of hydration of cement with selected retarders was evaluated. A thermocouple of type K and a portable data logger (TDS-303) from Tokyo Sokki Kenkyujo were used to monitor and record the temperature of each paste specimen for a period of not less than 24 hours which starts instantly after the mixing of the specimen; the experimental setup is shown diagrammatically in Figure 3.16.

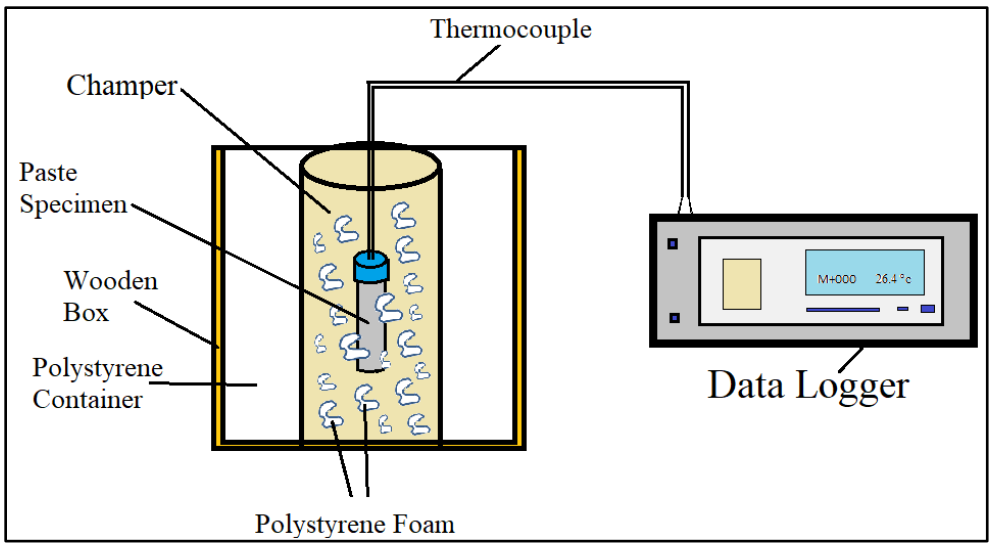

Figure 3.16: Setup of Heat of Hydration Test.

A plastic vial insulated with internal and external coatings of paraffin wax were used to incubate the specimens during the temperature monitoring. A 0.4 w/c ratio was used in all pastes. Immediately after a thorough mixing of cement, water and additive, the paste specimen was placed inside the vial. The weight of the vial was taken before and after the specimen's placement. For the purpose of comparison, all specimens were intended to have the same weight. Then, the thermocouple was embedded in the middle of the specimen and the vial was tightly closed with its screw cap. Next, the thermocouple was held in place with a duct tape. Figures 3.17 and 3.18 show the method used in preparing the specimen.

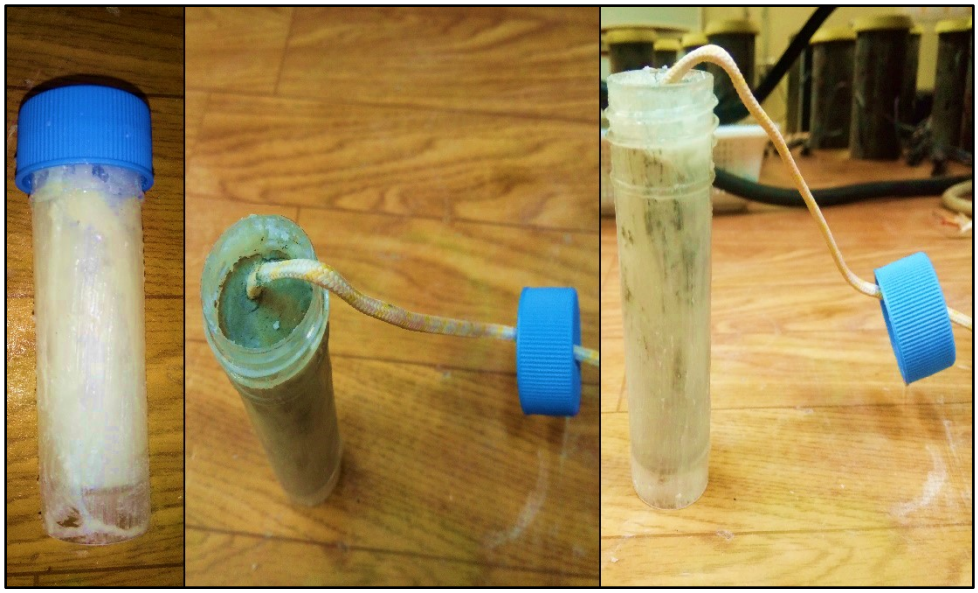

Figure 3.17: Preparation of the Specimen for Heat of Hydration Test (Part 1).

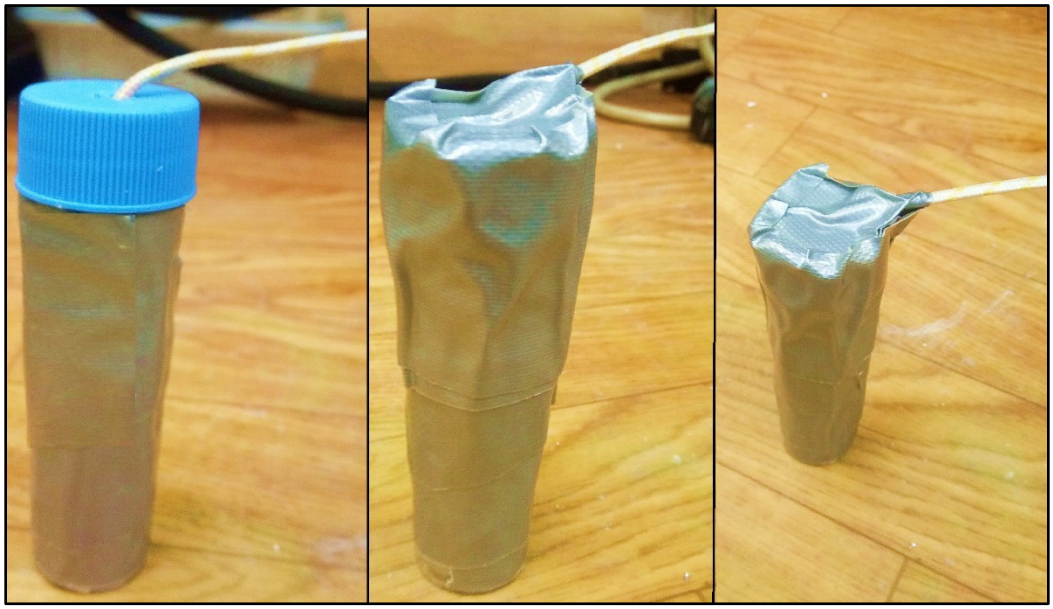

Figure 3.18: Preparation of the Specimen for Heat of Hydration Test (Part 2).

After specimen preparation, the vial was placed in a chamber which is already placed inside a thick polystyrene container. The polystyrene container is protected by a wooden box. Inside the chamber, the vial was surrounded by small chunks of polystyrene foam for further heat insulation. Figures 3.19 through 3.23 show the preventive measures used to minimize the heat exchange between the specimen and the surrounding environment.

Figure 3.19: Insulating Chamber

Figure 3.20: Polystyrene Foam.

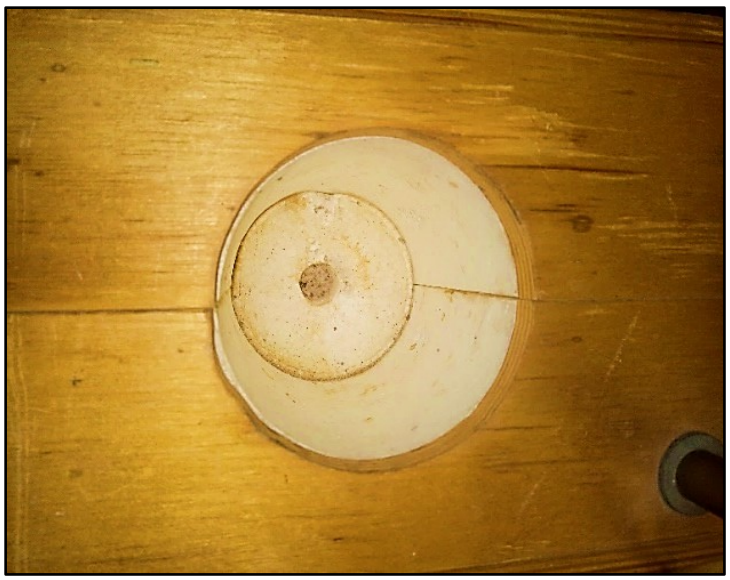

Figure 3.21: Polystyrene Container Inside a Wooden Box.

Figure 3.22: Placing of the Specimen Inside the Thermos and Confining with Polystyrene Foam.

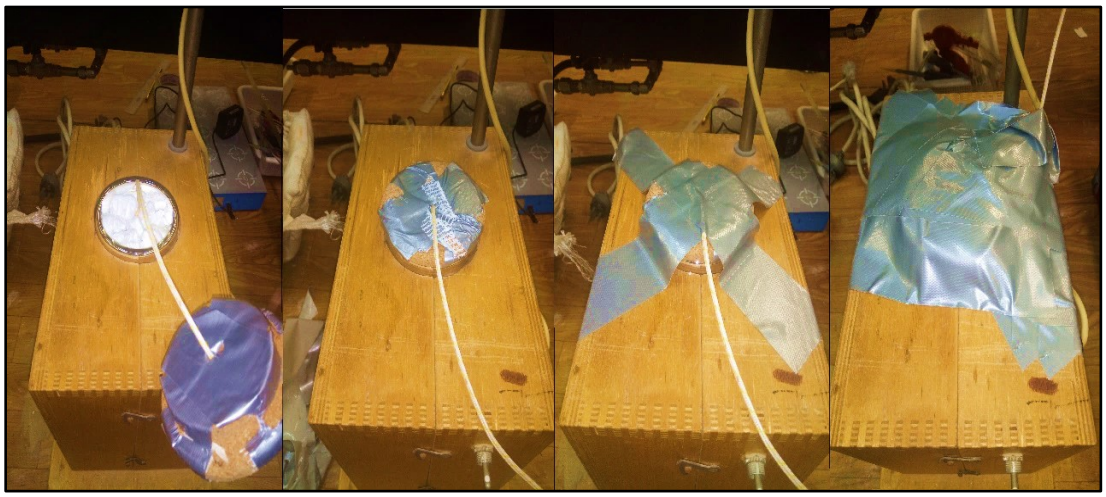

Figure 3.23: Sealing the Thermos Tightly with Lid and Duct Tape.

The thermocouple was embedded in the specimen and connected to the data logger and the temperature was measured continuously. For the sake of convenience, either 15-minute or 20-minute interval was selected to print the data during the whole duration of the test. Figure 3.24 shows the experimental setup used for measuring the heat of hydration and typical output is shown in Figure 3.25.

Figure 3.24: T Setup Used in Heat of Hydration Test.

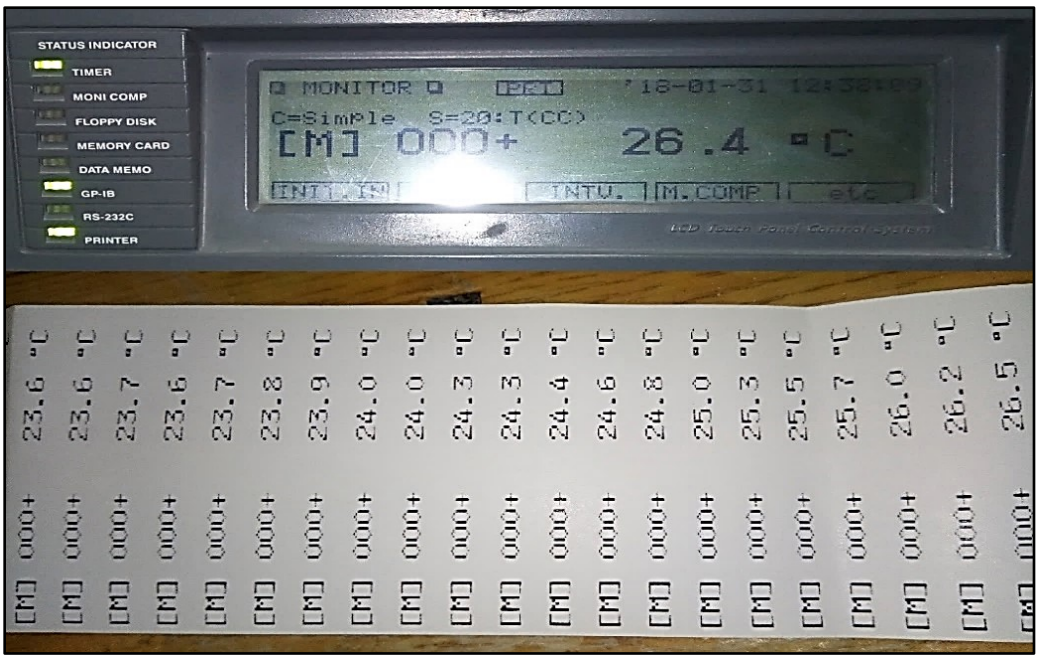

Figure 3.25: Temperature Readings Printed Using the Data Logger.

3.3.3 Slump Retention

According to ACI 116 [58], the term slump describes the consistency of fresh mixes of mortar or concrete directly after removing the slump cone containing the molded specimen. Slump retention is the variation of slump with time. In this study, the slump retention in freshly mixed concrete was measured using the method that is detailed in ASTM C143 [59]. The test was conducted on concrete containing each of the tested materials including a concrete specimen prepared only with OPC which will serve as a control specimen. Slump values in each specimen were measured at different time periods to determine the slump retention behavior over time. A mold which has the shape of a conical of frustum (slump cone) is filled with fresh concrete sample rodded in three layers, the mold's dimensions are shown in Figure 3.26. During the placement of the sample, the mold is held firmly on a nonabsorbent surface. Later, the mold is slowly lifted, allowing the concrete to freely slump. The length of the space between the initial and final levels of the displaced

center of the slumped concrete is determined and recorded to represent the slump value as shown in Figure 3.27. The test is conducted at different time periods until the concrete remarkably loses its workability.

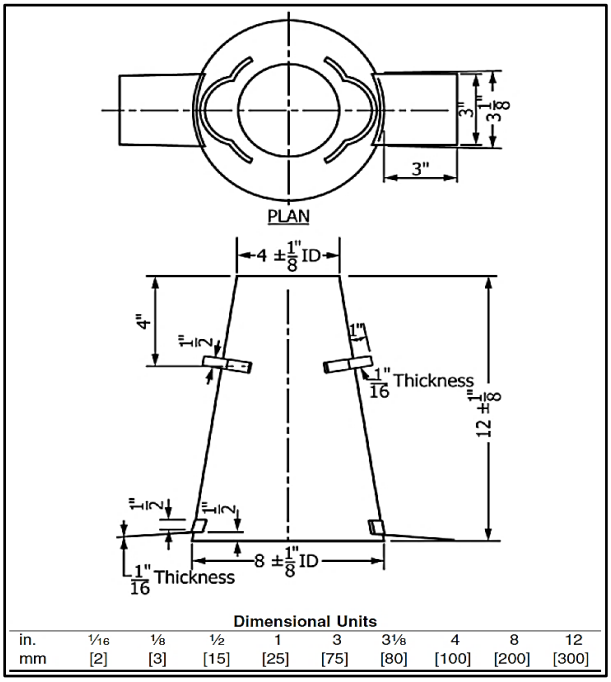

Figure 3.26: Dimensions of the Slump Cone [59].

Figure 3.27: Measuring of Slump in Concrete.

3.3.4 Compressive Strength

The compressive strength of the concrete specimens was examined according to ASTM C39 [60], after 3, 7, 14, 28 and 90 days of curing. At each age, three cylinders were sulfur-capped to obtain parallel and smooth surfaces for an even distribution of the force during the loading. Figure 3.28 shows a set of capped specimens. Next, the capped specimens were tested and the average strength in MPa was reported. An automatic, hydraulic compression testing machine (MATEST) was utilized to perform the compression test. Figure 3.29 shows the experimental setup.

Figure 3.28: Sulfur Capping for Cylindrical Concrete Specimens.

Figure 3.29: Compressive Strength Test.

3.3.5 Drying Shrinkage

As stated by ACI 116 [58], drying shrinkage can be defined as the reduction in either the volume or the length of the specimen due to loss of moisture. In this study, three mortar prisms, each of 25×25×285 mm, were used in determining the drying shrinkage for each mortar mixture in agreement with ASTM C596 [61] and ASTM C157 [62]. Figure 3.30 shows some molded shrinkage specimens, and Figure 3.31 shows some prisms during the drying period.

A portable data logger (TDS-301) from Tokyo Sokki Kenkyujo and an LVDT were used to measure the change of length of each prism over time. During the test, a frame with a smooth base was used to hold the specimen at a fixed vertical position, and a standard rod of a known length was used to set the reading of the data logger to zero. An initial reference reading of the length of each prism was taken at the end of the 28 days of submerged curing. Later, the drying shrinkage was measured at 4, 7, 14, 28, 56 and 90 days of air exposure

starting after the 28-day curing period. Figure 3.32 displays the setup used to monitor the drying shrinkage.

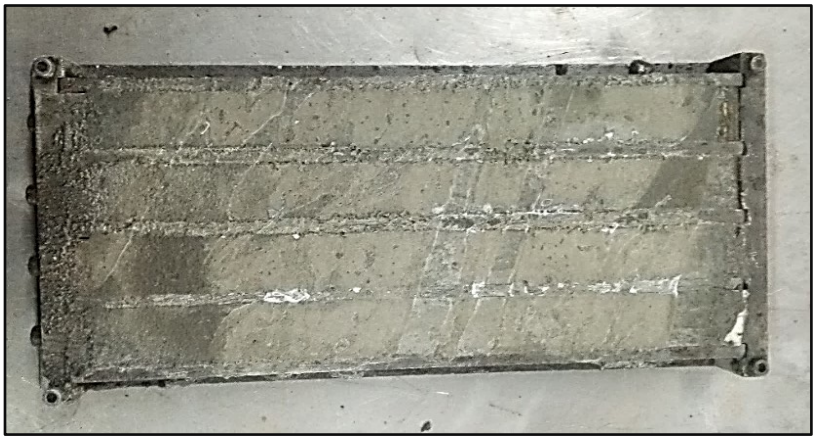

Figure 3.30: Drying-Shrinkage Molded Specimens.

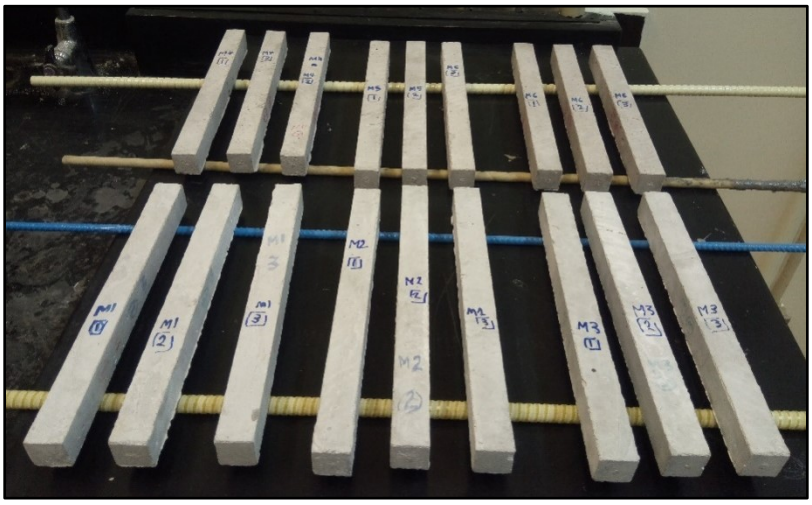

Figure 3.31: Drying-Shrinkage Specimens.

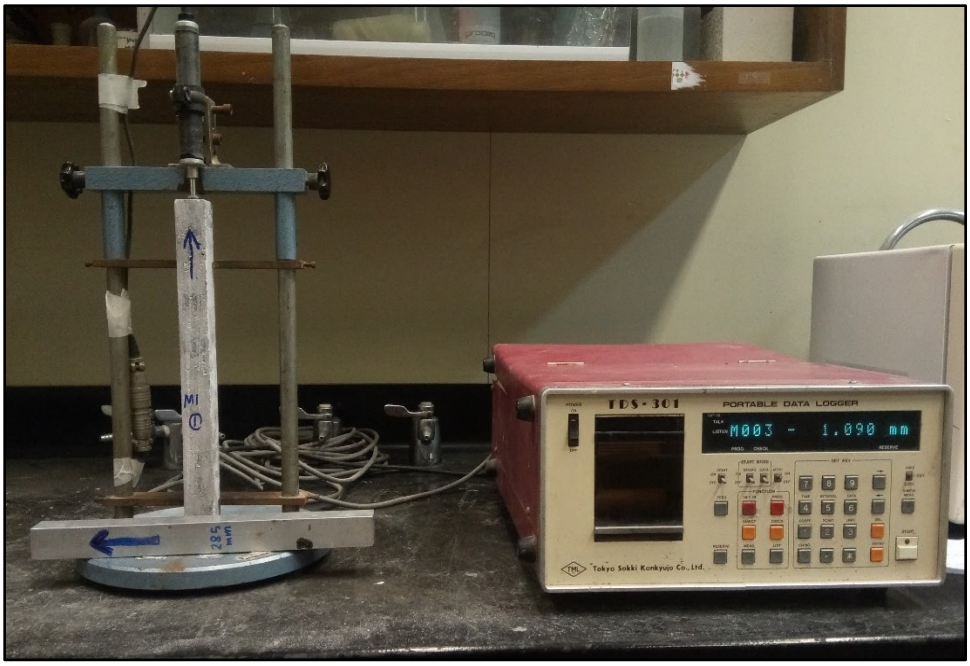

Figure 3.32: The Setup Used to Monitor the Drying Shrinkage.

3.3.6 Scanning Electron Microscopy (SEM)

SEM provides a unique capability for the description of the complex microstructure and constituents of the hardened cement paste and concrete. SEM produces images with a greater resolution and contrast than that of the light microscopy techniques. SEM can be used to identify the elements, composition, and phases of the tested substances including the quantities and the spatial distribution [63]. For thin specimens of the material, SEM provides high resolution images reaching a level of a few angstroms [64]. In this study, different paste specimens (cured for 28 days) were selected to be examined by SEM since changes in the microscopic morphology may take place.

After water curing, the hardened paste specimens were sealed in plastic bags, marked for identification and reserved until the testing time. On testing date, smooth thin slices were obtained from the interior of the hardened specimens to be tested. Before commencing the

test, the specimens were prepared as depicted in Figure 3.33 where a fine-grain sputtering of gold was applied over the slices of the examined pastes using a sputter coater (Cressington 108 auto) which is shown in Figure 3.34. Next, the specimens were placed in the scanning electron microscope (JOEL, JSM-6610LV) following the manufacturer's guidelines (Figure 3.35). A computer was used to select the surface to be scanned, to focus the electron beam and to control the magnification process, see Figure 3.36.

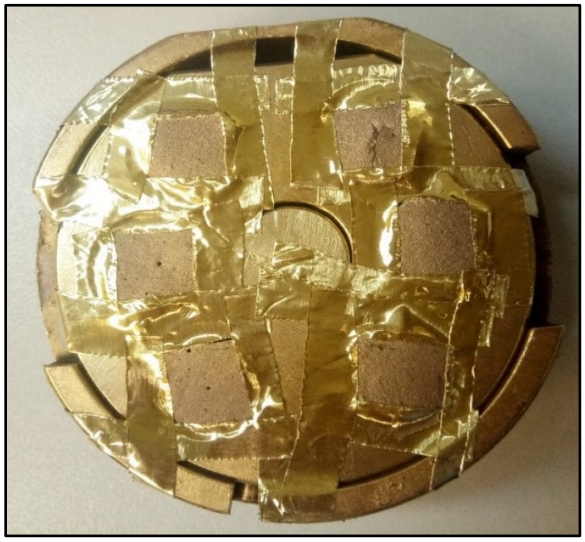

Figure 3.33: Specimens Prepared for SEM.

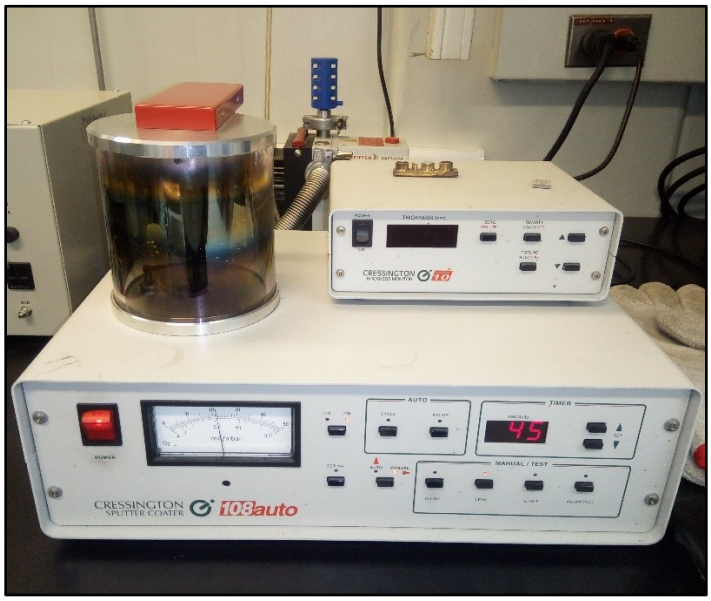

Figure 3.34: Sputter Coater for SEM.

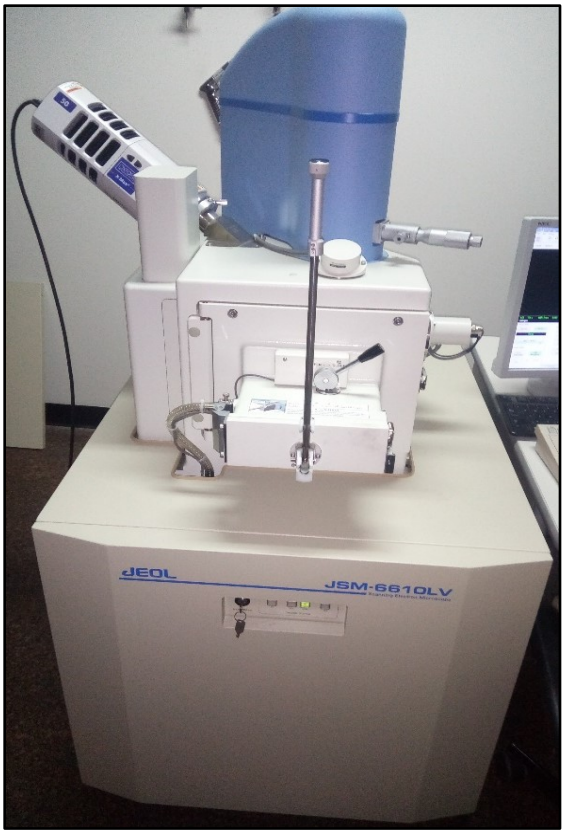

Figure 3.35: Scanning Electron Microscope.

Figure 3.36: Computer-Assisted SEM

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter demonstrates and discusses the results which were obtained through testing and experimentation. Details of mortar and concrete mix proportions are described in Section 3.2.

4.2 SETTING TIME

4.2.1 Effect of EAFD on Setting Time of Cement

Setting time of cement pastes incorporating EAFD is summarized in Table 4.1 and plotted in Figure 4.1. The dosages were varied from 0.5% to 6%, as an addition of cement. According to the data Table 4.1, there is a clear trend of a substantial increase in the setting time corresponding to an increase in the dosage of EAFD. The use of more than 3% EAFD resulted in a final setting time of more than 600 minutes.

Table 4.1: Setting Time with Varying Dosage of EAFD.

Dosage	IST (Minutes)	FST (Minutes)
0.5%	165	225
1%	180	240
2%	280	315
3%	440	465
4%	595	690
5%	875	970
6%	1075	1190

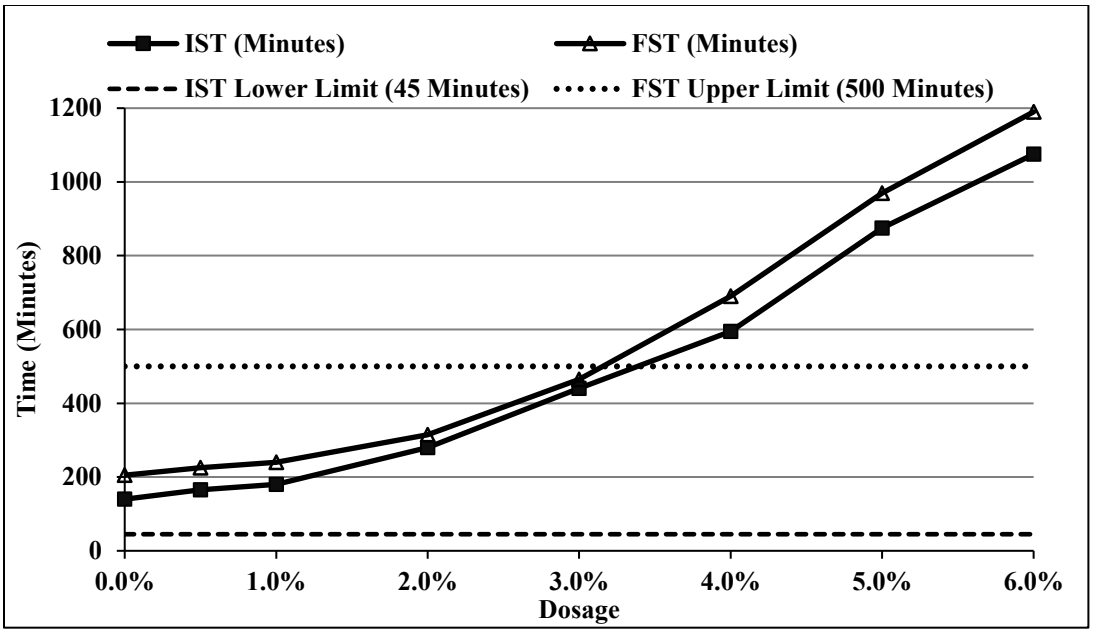

Figure 4.1: Setting Time with Varying Dosage of EAFD.

The increase in the setting time due to EAFD can be attributed to its content of zinc which is approximately 10%. Fares et al. [44] reported that zinc compounds in EAFD cause a retardation to the hydration reactions in concrete. As stated by Arliguie and Grandet [66], the presence of Zn in the mix creates an amorphous coat of $Zn(OH)_2$ over the anhydrous particles of cement which temporarily hinders the hydration of C_3S phase. Later, the hydration process is restored through the transformation of $Zn(OH)_2$ into crystalline calcium zinc hydroxide when the concentration of Ca^{2+} and OH^- is enough in the pore solution.

4.2.2 Effect of Thermally Treated EAFD on Setting Time of Cement

Setting time of cement pastes containing selected dosages of T-EAFD (thermally treated at 800 °C for 2 hours) are provided in Table 4.2 and plotted in Figure 4.2. The dosage of treated EAFD was varied from 5% to 40%.

Table 4.2: Setting Time After the Addition of T-EAFD

Dosage	IST (Minutes)	FST (Minutes)
5%	150	210
10%	155	215
30%	180	220
40%	205	260

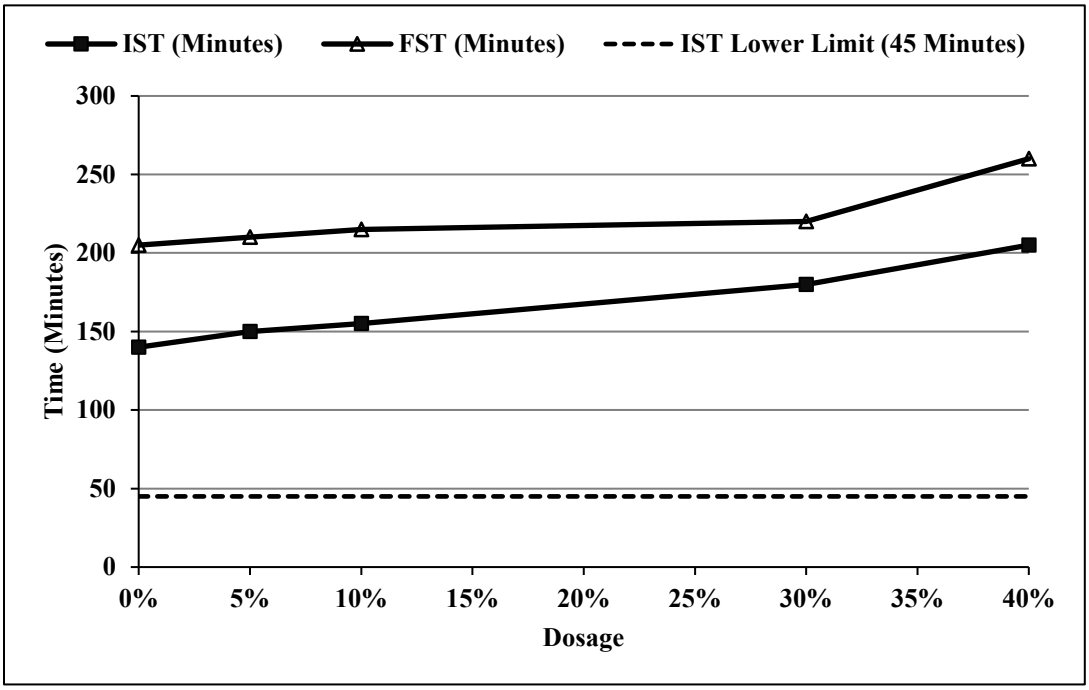

Figure 4.2: Setting Time with Varying Dosage of T-EAFD.

As indicated by the data in Table 4.2, thermally treated EAFD had a marginal effect on increasing the setting time when it is added to the cement in low percentages. However, the use of higher percentages (30% and 40% EAFD) prolonged the setting time significantly. The final setting time was less than 500 minutes. This extension of setting time is due to the remnant of the content of zinc in EAFD after the thermal treatment.

Lee and Song [67] reported that when EAFD is heated, volatilization of metals including Zn takes place. In that research, it was found that volatilization of Zn starts rapidly at 600 °C and that the increase in the rate of volatilization is proportional to the temperature, it was also reported that the volatilization of Zn in EAFD is complete at 1000 °C.

As mentioned earlier, after heating of EAFD at a temperature of 800 °C for 2 hours, the quantity of Zn will be significantly reduced. Therefore, a larger quantity of EAFD is needed to achieve a considerable increase in the setting time.

4.2.3 Effect of Oil Ash on Setting Time of Cement

Setting time of cement pastes incorporating oil ash (varying from 0.5% to 3%), are given in Table 4.3 and plotted in Figure 4.3.

Table 4.3: Setting Time After the addition Oil Ash.

Dosage	IST (Minutes)	FST (Minutes)
0.5%	145	220
1%	170	235
1.5%	180	245
2%	210	290
3%	235	315

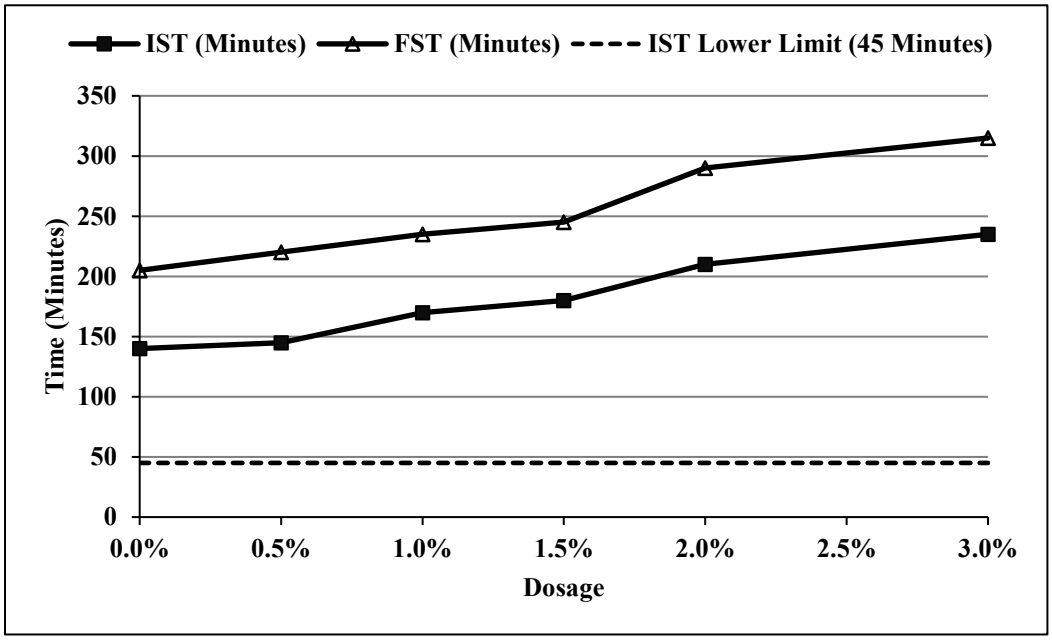

Figure 4.3: Setting Time with Varying Dosage of Oil Ash.

The use of 1% and 1.5% oil ash improved the setting time. Moreover, there was a satisfactory extension of the setting time with the use of 2% and 3% oil ash and the final setting time was less than 500 minutes.

The ability of oil ash to extend the setting time was confirmed by Camilleri et al. [45]. In that study, it was reported that oil ash caused an increase in the setting time of different tested mortars through formation of ettringite coating over the surfaces of cement particles soon after mixing, due to the presence of sulfur (sulfate) in oil ash. Ettringite coating reduces the reaction between water and unhydrated cement.

4.2.4 Effect of Commercial Retarders on Setting Time of Cement

Setting time of cement pastes incorporating Conplast RP264® and Sika Retarder 21® are provided in Tables 4.4 and 4.5 and plotted in Figures 4.4 and 4.5 respectively. Based on the recommendations of manufacturers, Conplast RP264 was used in dosages varying from 0.35% to 0.95%, and Sika Retarders was used in dosages ranges between 0.1% to 1.2%.

Table 4.4: Setting Time with Varying Dosage of Conplast RP264®.

Dosage	IST (Minutes)	FST (Minutes)
0.35%	405	450
0.40%	435	510
0.65%	750	810
0.95%	875	1080

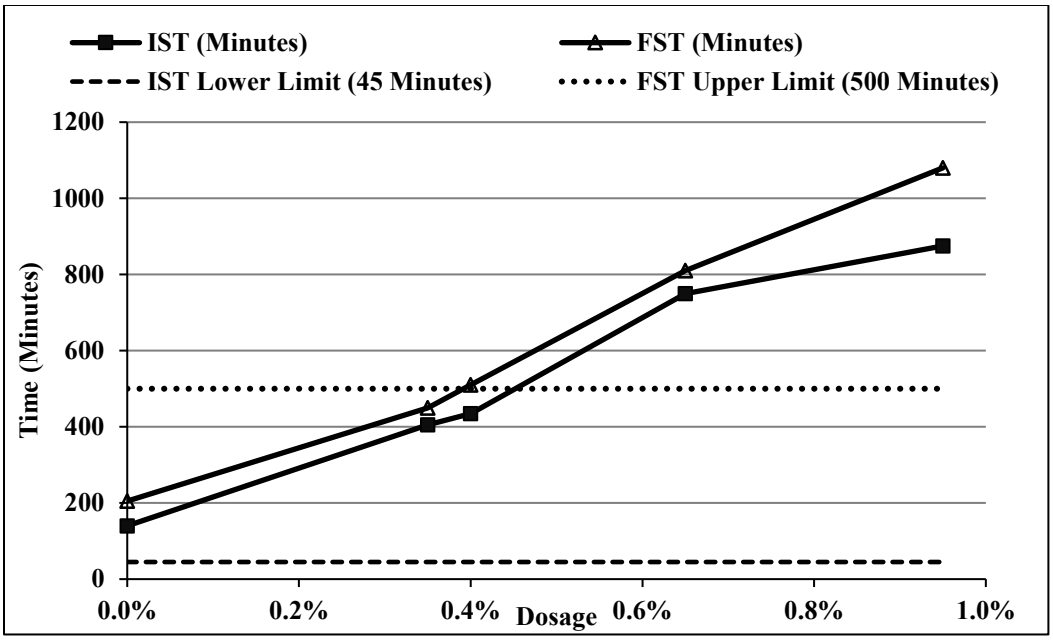

Figure 4.4: Setting Time with Varying Dosage of Conplast RP264®.

According to the data Table 4.4, the addition Conplast RP264 extended the time of set. The retardation ability of Conplast RP264 is consistent with the previous related literature. The product datasheet, provided by the manufacturer, states that Conplast RP264 is based on lignosulfonate. According to Ramachandran and Feldman [68], the retardation effect of lignosulfonates refers to the great adsorption influences on tricalcium aluminate phase. Young [17] suggested that the retardation effect of lignosulfonate-based admixtures can be ascribed to the adsorption of the organic matter onto the surface of $Ca(OH)_2$ nuclei which imposes a retardation to the hydration of C_3S.

Table 4.5: Setting Time with Varying Dosage of Sika Retarder 21®.

Dosage	IST (Minutes)	FST (Minutes)
0.1%	170	225
0.2%	185	240
0.8%	450	495
1.2%	470	520

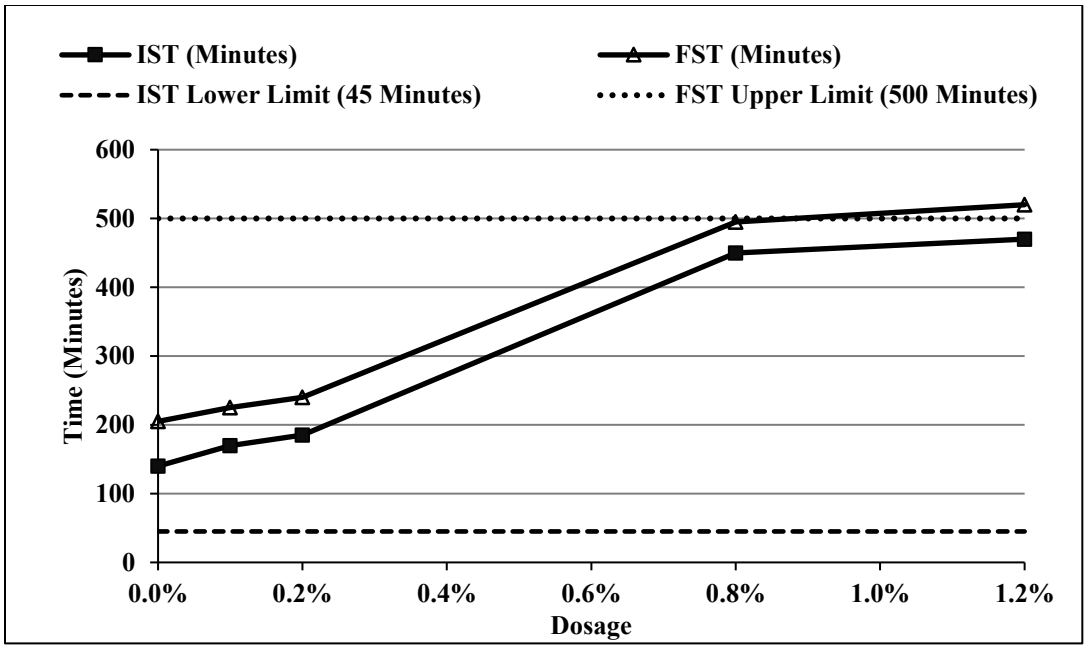

Figure 4.5: Setting Time with Varying Dosage of Sika Retarder 21®.

As shown in Table 4.5, Sika retarder 21 increased the setting time up to a dosage of 1.2%. However, the rate of increase seems to start decreasing after a dosage of 0.8%. The product datasheet provided by the manufacturer states that Sika retarder 21 is based on phosphate. According to Ramachandran [15], the majority of phosphates have a retardation influence on the setting time. The mechanism of retardation is based on the precipitation of Ca-Phosphates that is resulted from the adsorption of phosphate ion ($PO4^{3-}$) either on the surface of the clinker phase or on the first hydration products. Basically, Ca-Phosphates retard the hydration reaction. Monteiro [69] confirms that phosphates are frequently used as the retarding ingredient in many commercial retarding admixtures.

4.2.5 Effect of Chemical Retarders on Setting Time of Cement

Setting time of cement pastes with varying dosages of sugar, ZnO and sodium gluconate is presented in Table 4.6 through Table 4.8 and plotted in Figure 4.6 through 4.8, respectively.

Table 4.6: Setting Time with Varying Dosage of Sugar.

Dosage	IST (Minutes)	FST (Minutes)
0.01%	200	255
0.02%	275	365
0.03%	370	395
0.04%	545	590
0.05%	960	1060

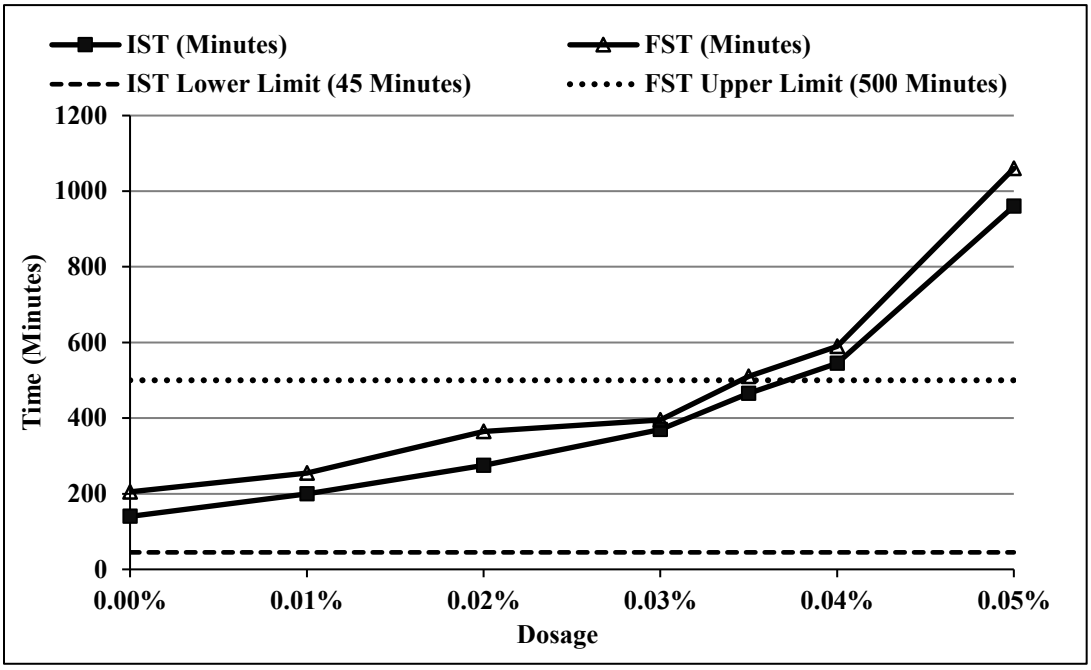

Figure 4.6: Setting Time with Varying Dosage of Sugar.

It is clear from the data in Table 4.6 that increasing the dosage of sugar causes a notable increase in the setting time, which agrees with the available literature [18]–[23]. Other studies tried to identify the mechanism of retardation due to sugar addition. According to Thomas and Birchall [70], sugar is adhered to the outside surface of hydration products and poisons the CH and C-S-H gel. Thus, it causes a retardation of the hydration reaction.

During the trial experiments, in other specimens which are not mentioned in Table 4.6, increasing sugar dosage from 0.05% to 0.1%, 0.18% and 0.25% produced setting time of more than 1000 minutes. However, the use of 0.8% sugar significantly reduced the setting time below that of OPC which means that sugar starts to work as a set accelerator above a

certain dosage. This was quite anticipated according to the literature. Meyer and Perenchio [71] reported that sugar is a strong retarder for C_3S phase. However, at higher sugar dosages, the hydration of C_3S is severely hindered which lowers the concentration of Ca^{+2} in the solution. Thus, the solubility and hydration of C_3A are increased leading to a rapid setting of cement, but this process does not significantly increase the early strength since the hydration of C_3S is already impeded.

Table 4.7: Setting Time with Varying Dosage of ZnO.

Dosage	IST (Minutes)	FST (Minutes)
0.02%	200	260
0.05%	290	330
0.08%	335	430
0.09%	470	550
0.15%	1140	1265

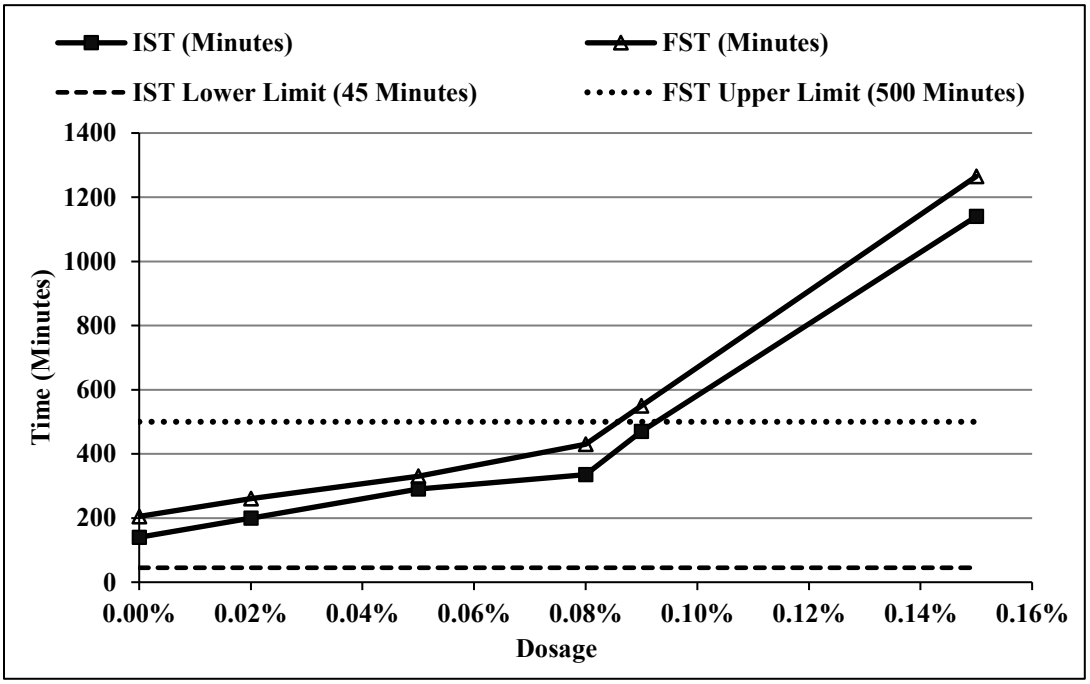

Figure 4.7: Setting Time with Varying Dosage of ZnO.

There is an important increase in the setting time with an increase in the dosage of ZnO as shown in Table 4.7. During the trials, in other specimens which are not given in Table 4.7,

increasing zinc oxide dosage from 0.08% to 0.5% and 1% produced final setting time of more than 1300 minutes.

Cheeseman et al. [72] reported that the presence of Zn promotes the retardation of hydration reaction through the formation of a coat of $CaZn_2(OH)_6.H_2O$ around cement clinker particles.

Table 4.8: Setting Time with Varying Dosage of Sodium Gluconate.

Dosage	IST (Minutes)	FST (Minutes)
0.01%	160	220
0.02%	185	245
0.05%	215	275
0.06%	210	270
0.08%	155	220

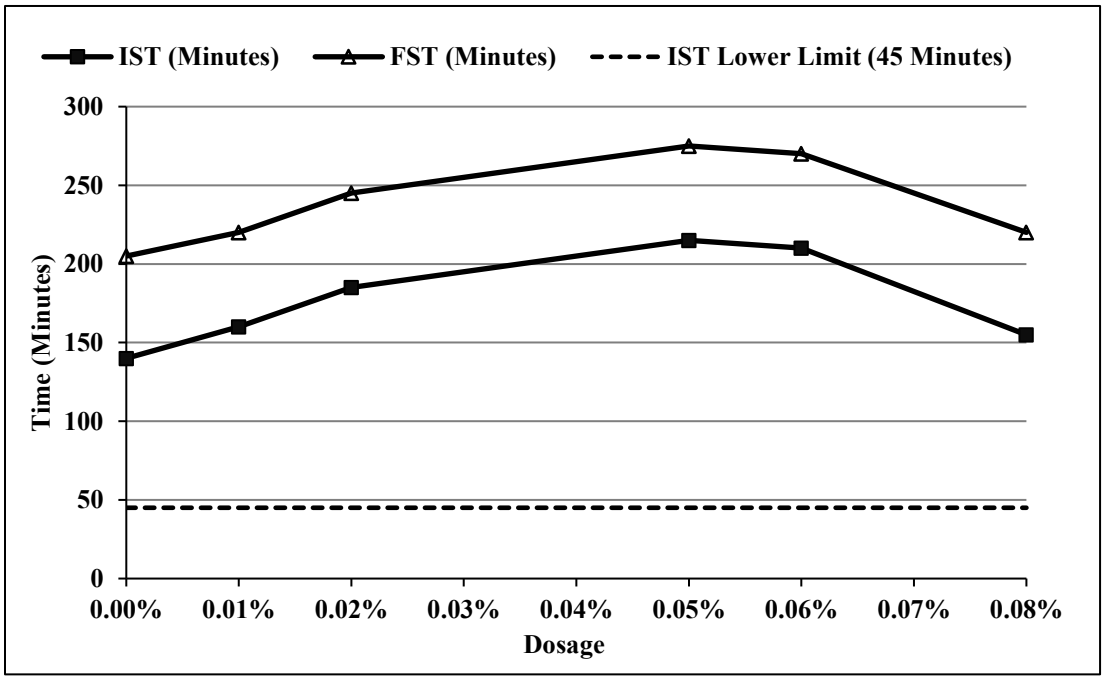

Figure 4.8: Setting Time with Varying Dosage of Sodium Gluconate.

The final setting time due to the addition of sodium gluconate was less than 500 minutes (Table 4.8).The setting time started to increase gradually corresponding to the increase in

the quantity of sodium gluconate up to 0.05% sodium gluconate. However, setting time of paste with 0.06 and 0.08% sodium gluconate was below that of 0.05% sodium gluconate.

According to Li et al. [28], sodium gluconate is adsorbed onto C_3S and C_2S surfaces. Thus, it causes a retardation to the cement setting.

Table 4.9 summarizes effects of selected dosages of retarders on the initial and final setting time of cement. The dosages shown below were selected based on the criteria that the final setting time is to be less than 500 minutes. The dosages were used in mortar and concrete mixtures to examine other properties.

Table 4.9: Effects of Selected Dosages of Retarders on the Initial and Final Setting Time of Cement.

Material	Dosage	IST (Minutes)	FST (Minutes)
OPC	--	140	205
EAFD	0.5%	165	205
	1%	180	240
	2%	280	315
	3%	440	465
T-EAFD	30%	180	220
	40%	205	260
Oil Ash	1.5%	180	245
	2%	210	290
	3%	235	315
Conplast RP264®	0.35%	405	450
Sika Retarder 21®	0.80%	450	495
Sugar	0.03%	370	395
ZnO	0.08%	335	430
Sodium Gluconate	0.05%	215	275

Based on setting time results, the most effective cement set retarders were Conplast RP264®, EAFD, sugar and ZnO. Each addition of these materials was associated with a substantial increase in the setting time. EAFD, Conplast RP264®, sugar and ZnO can be used to extend the initial setting time of OPC steadily from 140 min up to more than 900 minutes as shown in previous setting time figures. The addition of Sika Retarder 21® was

effective in prolonging the setting time of cement, but the maximum initial setting time was in the about 470 minutes as shown in Figure 4.5. The initial setting time of 2 and 3% oil ash was 210 and 235 min, respectively. Sodium gluconate was less effective in extending the setting time. A maximum initial setting time of around 215 min was attained at a dosage of 0.05%, further increase in the dosage began to gradually reduce the setting time. Thermally treated EAFD can only extend the setting time remarkably at high dosages, it extended the initial setting time of OPC from 140 min to 180 and 205 min by dosages of 30 and 40%, respectively.

4.3 HEAT OF HYDRATION

4.3.1 Effect of EAFD on Heat of Hydration

The evolution of heat of hydration of cement containing EAFD is presented in Figure 4.9.

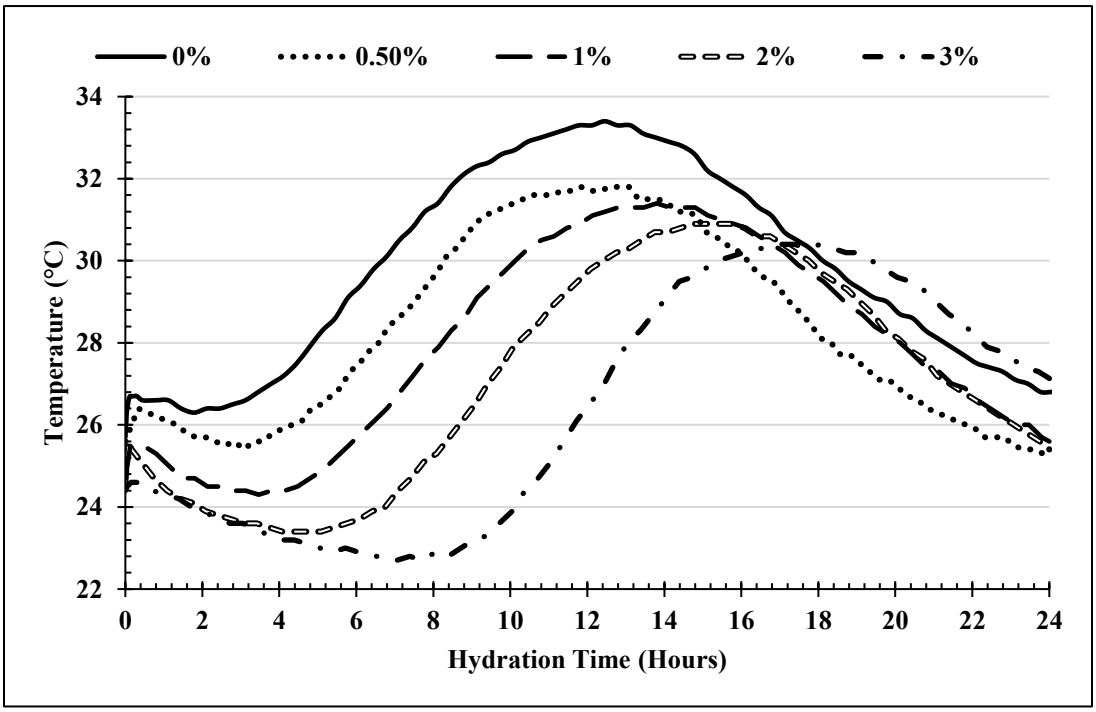

Figure 4.9: Influence of EAFD on the Evolution of Temperature Due to Hydration of Cement.

Values of dormant period, peak temperature (°C) and time of occurrence of the main temperature peak are presented in Table 4.10. As shown in the table, all dosages of EAFD prolonged the dormant period and postponed the time of occurrence of the main temperature peak. Values of peak temperature also decrease as the addion of EAFD increases.

Table 4.10: Dormant Period, Peak Temperature and Time of Appearance of Peak Temperature after the Addition of EAFD.

Dosage	Dormant Period (Hours)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
0%	2.3	33.4	12.4
0.5%	2.5	31.8	13
1%	3.7	31.4	13.8
2%	5	30.9	15.3
3%	7.3	30.4	17.4

4.3.2 Effect of Thermally Treated EAFD on Heat of Hydration

The evolution of heat of hydration of cement containing T-EAFD is presented in Figure 4.10.

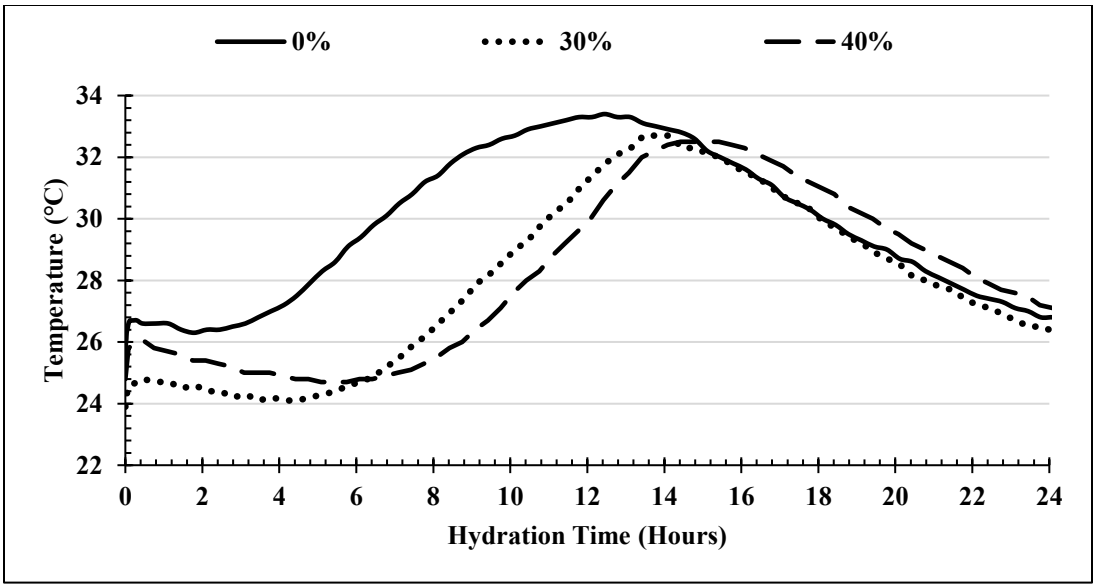

Figure 4.10: Influence of T-EAFD on the Evolution of Temperature Due to Hydration of Cement.

Table 4.11 summarizes values of dormant period, peak temperature (°C) and time of occurrence of the main temperature peak. The data showed that T-EAFD extended the dormant period and postponed the time of appearance of the main temperature peak. Values of peak temperature slightly decreased with the addtion of T-EAFD.

Table 4.11: Dormant Period, Peak Temperature and Time of Appearance of Peak Temperature After the Addition of T-EAFD.

Dosage	Dormant Period (Hours)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
0%	2.3	33.4	12.4
30%	3.7	32.7	14
40%	5	32.5	15

4.3.3 Effect of Oil Ash on Heat of Hydration

The evolution of heat of hydration of cement containing oil ash is presented in Figure 4.11.

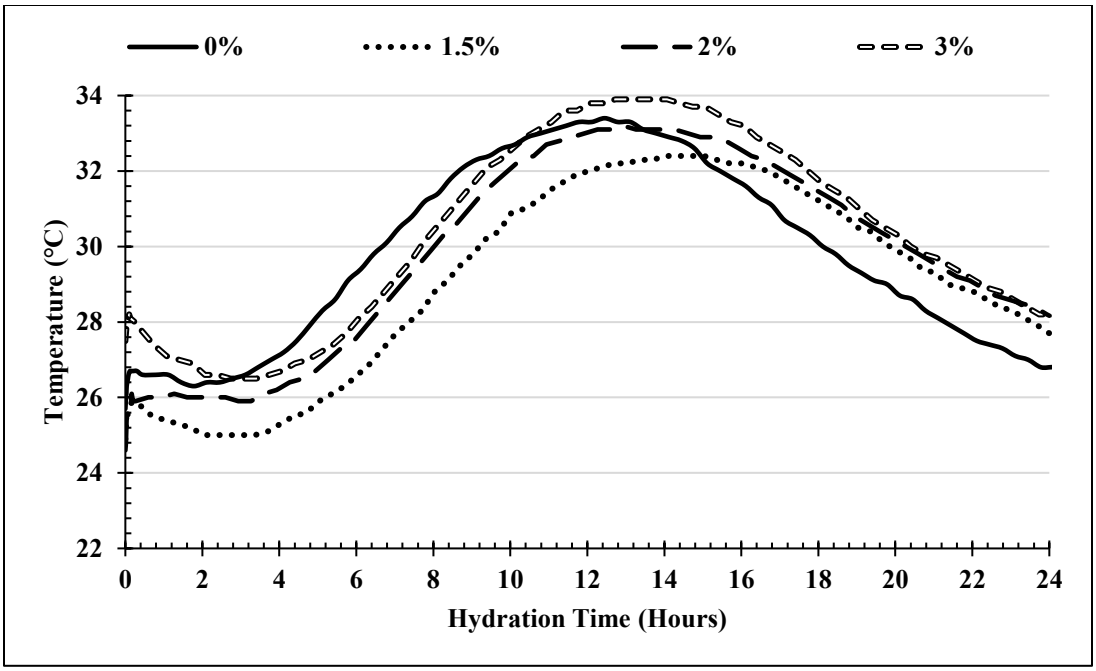

Figure 4.11: Influence of Oil Ash on the Evolution of Temperature Due to Hydration of Cement.

Values of dormant period, peak temperature (°C) and time of occurrence of the main temperature peak are given in Table 4.12. As shown in the table, oil ash slightly prolonged the dormant period and postponed the time of occurrence of the main temperature peak. Values of peak temperature of 1.5 and 2% oil ash were below that of 0% oil ash. However, 3% oil ash caused a marginal increase in the value of main temperature peak.

Table 4.12: Dormant Period, Peak Temperature and Time of Appearance of Peak Temperature After the Addition of Oil Ash.

Dosage	Dormant Period (Hours)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
0%	2.3	33.4	12.4
1.5%	3	32.4	14.5
2%	3.1	33.2	13.6
3%	3.3	33.9	13.4

4.3.4 Effect of Commercial Retarders on Heat of Hydration

The effect of 0.35% Conplast RP264® and 0.8% Sika Retarder 21® on the evolution of temperature due to the hydration of cement is plotted in Figure 4.12.

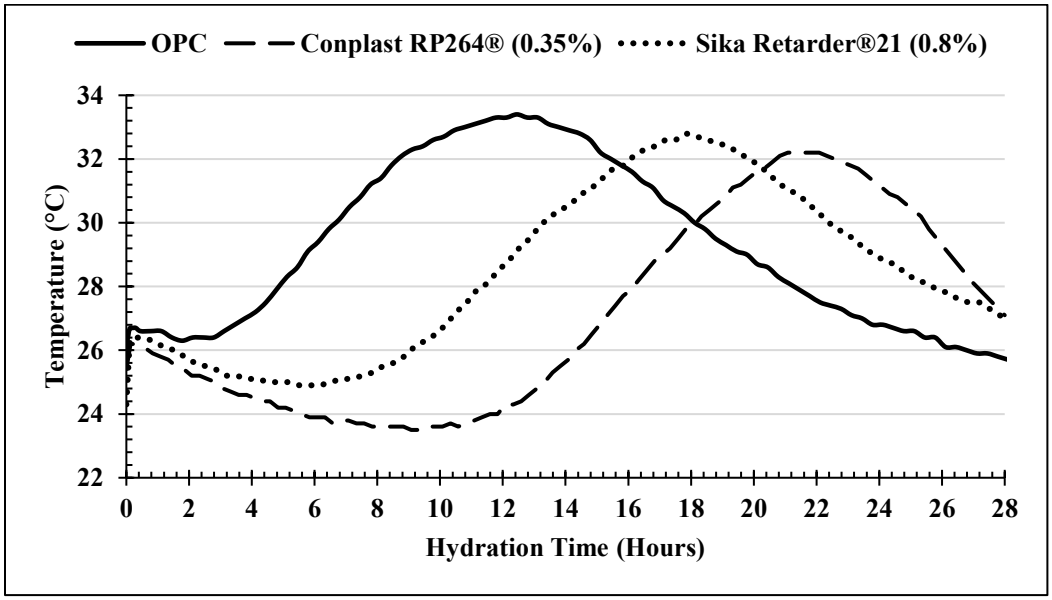

Figure 4.12: Influence of Commercial Retarders on the Evolution of Temperature Due to Hydration of Cement.

Table 4.13 gives values of dormant period, peak temperature (°C) and time of occurrence of the main temperature peak upon the addition of both commercial retarders. Conplast RP264 hindered the evolution of heat of hydration of cement for a longer time compared to Sika Retarder 21.

Table 4.13: Dormant Period, Peak Temperature and Time of Appearance of Peak Temperature After the Addition of Commercial Retarders.

Material	Dosage	Dormant Period (Hours)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
OPC	--	2.3	33.4	12.4
Conplast RP264®	0.35%	8.8	32.2	21
Sika Retarder 21®	0.80%	6	32.8	17.8

4.3.5 Effect of Chemical Retarders on Heat of Hydration

The Effect of 0.03% sugar, 0.08% ZnO and 0.05% sodium gluconate on the evolution of temperature due to the hydration of cement is plotted in Figure 4.13.

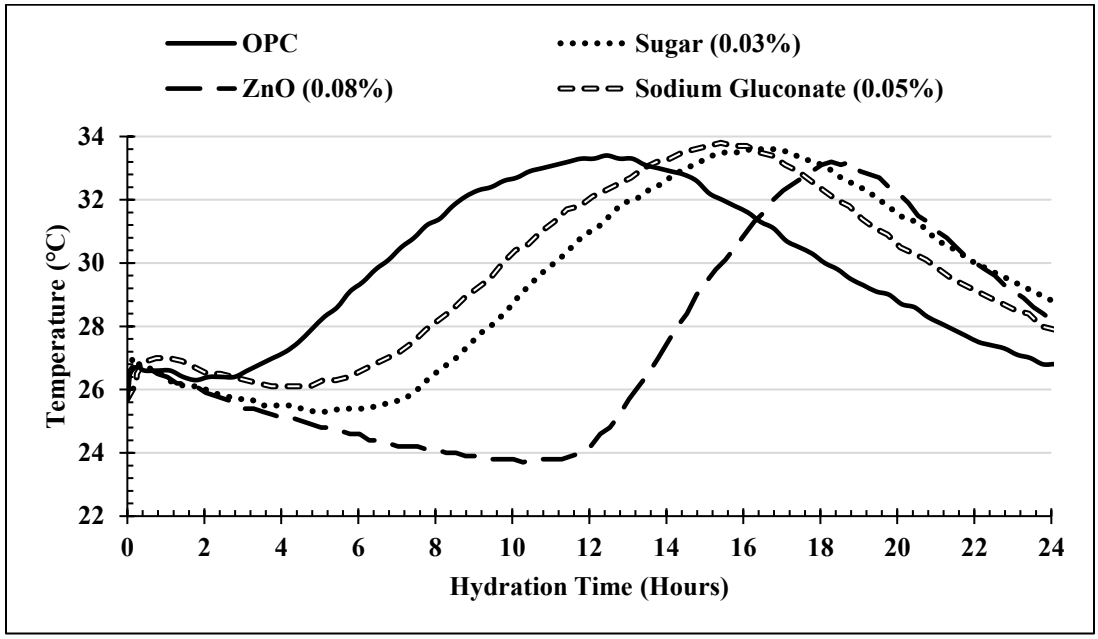

Figure 4.13: Influence of Chemical Retarders on the Evolution of Temperature Due to Hydration of Cement.

Table 4.14 gives values of dormant period, peak temperature (°C) and time of occurrence of the main temperature peak after using chemical retarders. ZnO exhibited the highest capacity to hinder the evolution of heat amongst the used chemical retarders. ZnO and sodium gluconate caused a marginal increase in the value of peak temperature.

Table 4.14: Dormant Period, Peak Temperature and Time of Appearance of Peak Temperature After the Addition of Chemical Retarders.

Material	Dosage	Dormant Period (Hours)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
OPC	--	2.3	33.4	12.4
Sugar	0.03%	6	33.6	16.5
ZnO	0.08%	10.8	33.2	18.3
Sodium Gluconate	0.05%	3.67	33.8	15.3

Figure 4.14 presents a summary of the effects of retarders on dormant period of cement hydration. A mathematical relationship between dormant period and initial setting time was developed as shown in Figure 4.15. This relationship can be used to predict the initial setting time using heat of hydration results.

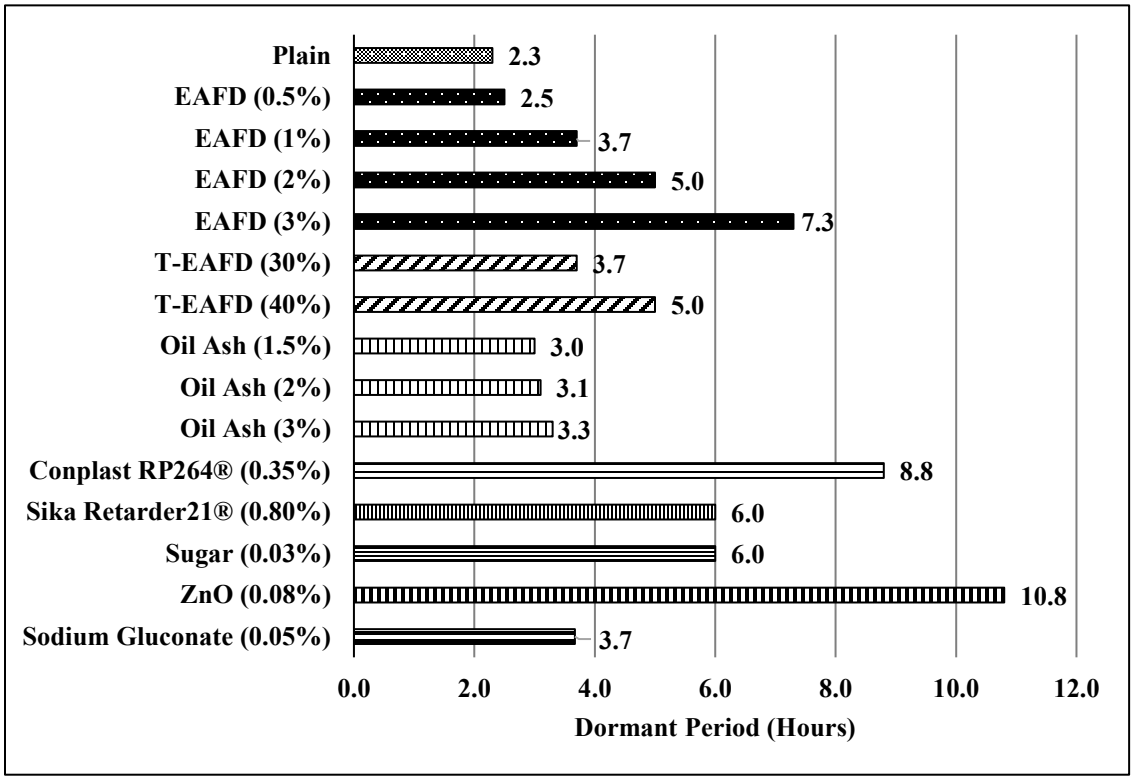

Figure 4.14: Effect of Retarders on Dormant Period.

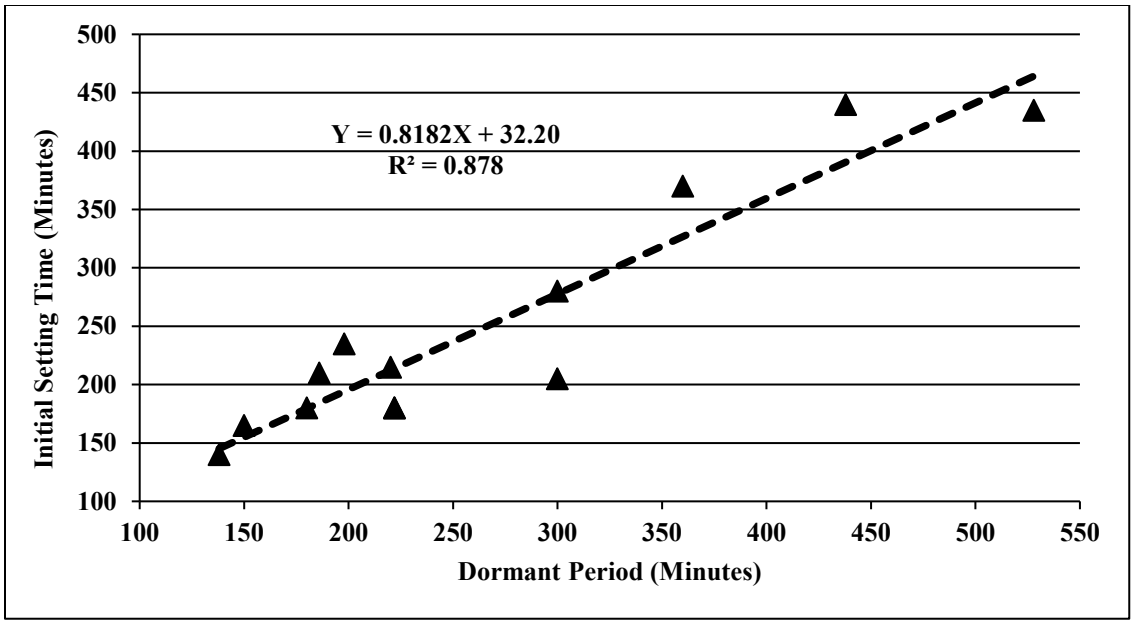

Figure 4.15: Relationship Between Dormant Period and Initial Setting Time.

Figure 4.16 presents a summary of the effects of retarders on time to peak temperature due to cement hydration. A mathematical relationship between time to peak temperature and final setting time was developed as shown in Figure 4.17. This relationship can be used to predict the final setting time using heat of hydration results.

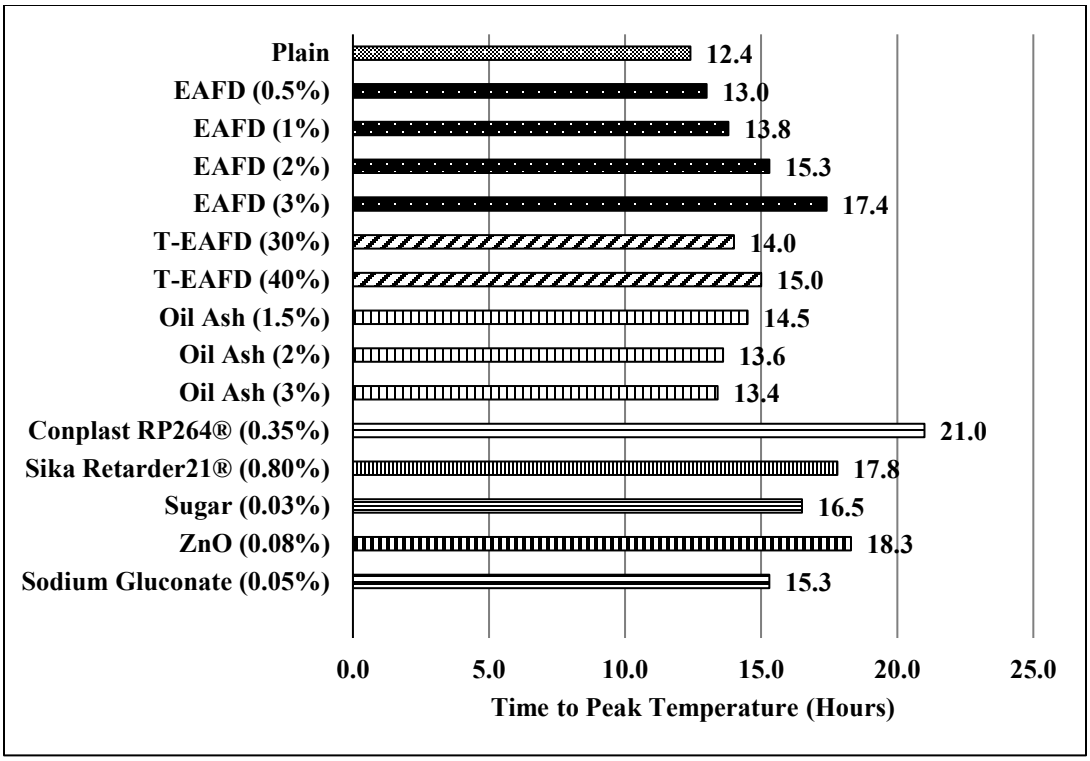

Figure 4.16: Effect of Retarders on Time to Peak Temperature.

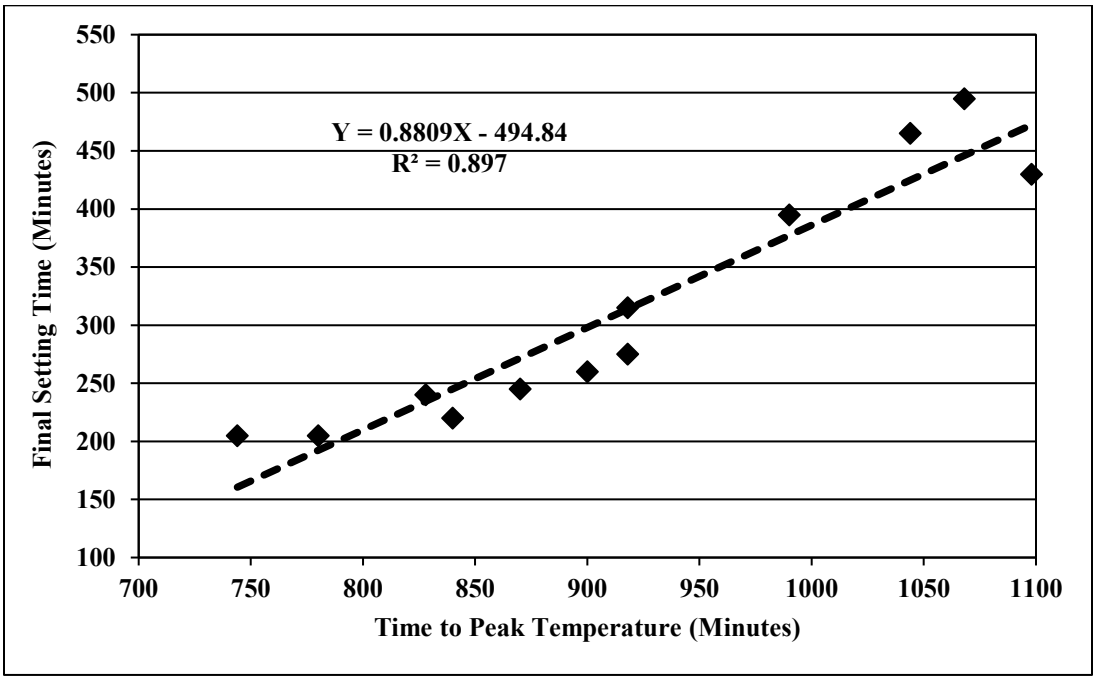

Figure 4.17: Relationship Between Time to Peak Temperature and Final Setting Time.

4.4 SLUMP RETENTION

4.4.1 Effect of EAFD on Slump Retention

The variation of slump with time in concrete mixes incorporating variable dosages of EAFD is presented in Figure 4.18.

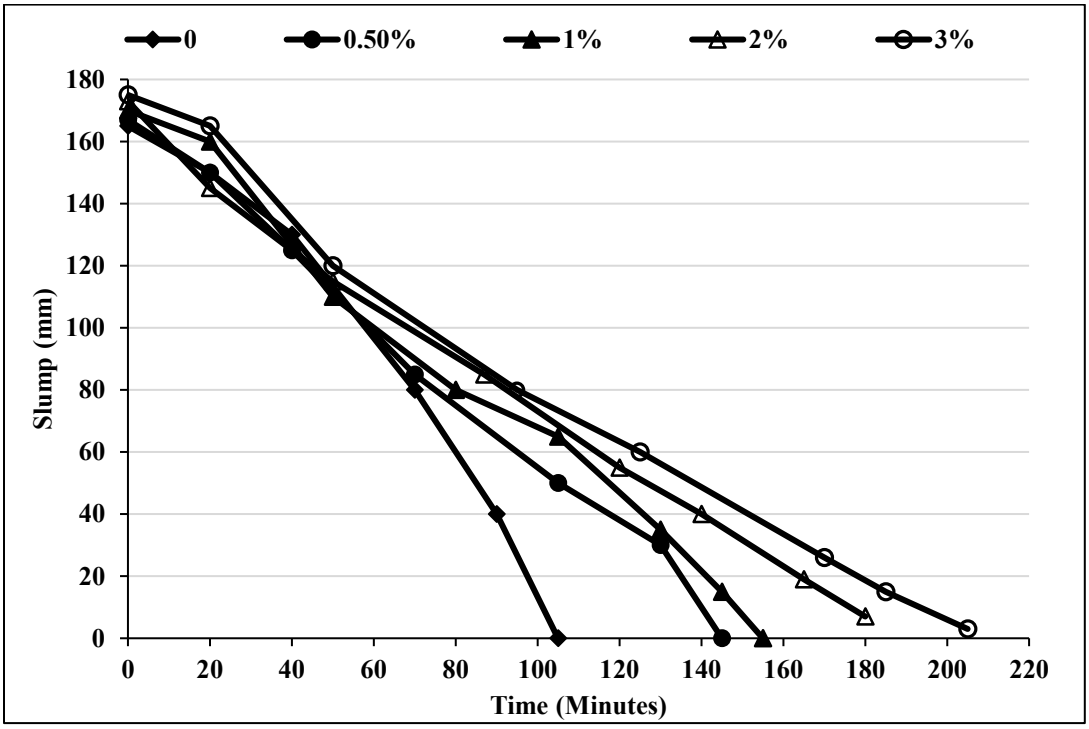

Figure 4.18: Slump Retention in Concrete Mixes with Varying Dosage of EAFD.

The influence of EAFD on initial slump values and slump retention time is given in Table 4.15.

Table 4.15: Effect of EAFD on Slump Retention in Concrete.

Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
0%	165	105
0.5%	167	145
1%	170	155
2%	173	180
3%	175	205

According to the data in Table 4.15, the incorporation of EAFD increased the initial slump and improved the slump retention time in concrete. The effect was remarkable at 2 and 3% EAFD. The improvement of slump retention time can be ascribed to the retardation effect of EAFD.

4.4.2 Effect of Thermally Treated EAFD on Slump Retention

Effect of thermally treated EAFD (T-EAFD) on the variation of slump with time in concrete is shown in Figure 4.19.

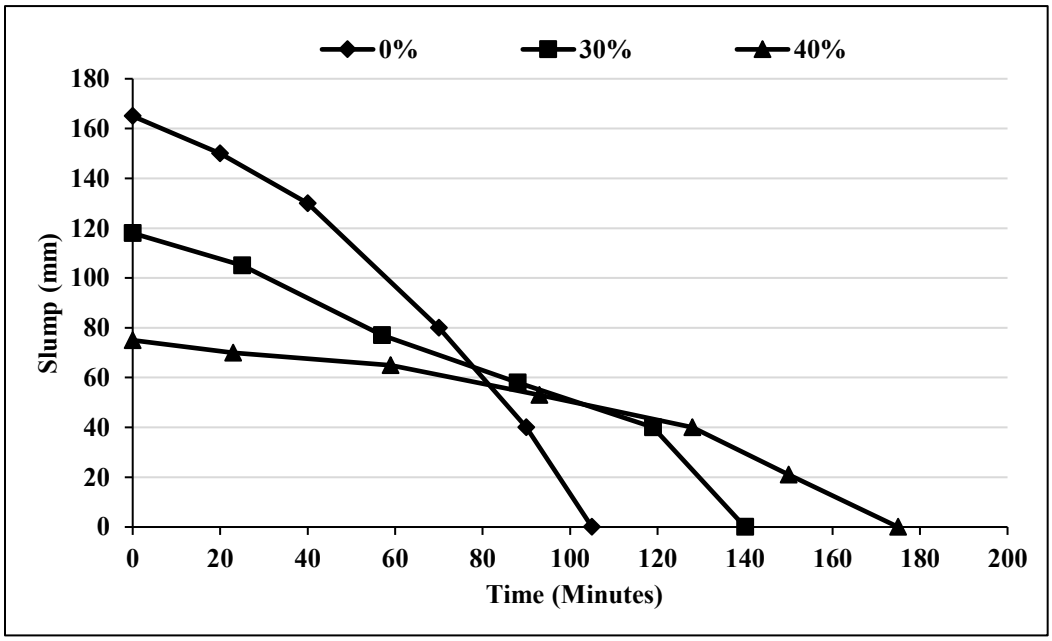

Figure 4.19: Slump Retention in Concrete Mixes with Varying Dosage of T-EAFD.

Table 4.16 shows initial slump values and the time required for concrete to lose its workability after the use of T-EAFD.

Table 4.16: Effect of T-EAFD on Slump Retention in Concrete.

Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
0%	165	105
30%	118	140
40%	75	175

According to the data in Table 4.16, the addition of 30% and 40% T-EAFD retained the slump for a longer period. However, the initial slump values of 30 and 40% T-EAFD concrete was less than that of plain concrete. The reduced slump can be attributed to the increase in the content of fine materials in the mix due to the addition of large quantity of T-EAFD.

4.4.3 Effect of Oil Ash on Slump Retention

The variation of slump with time in concrete mixes incorporating oil ash is plotted in Figure 4.20.

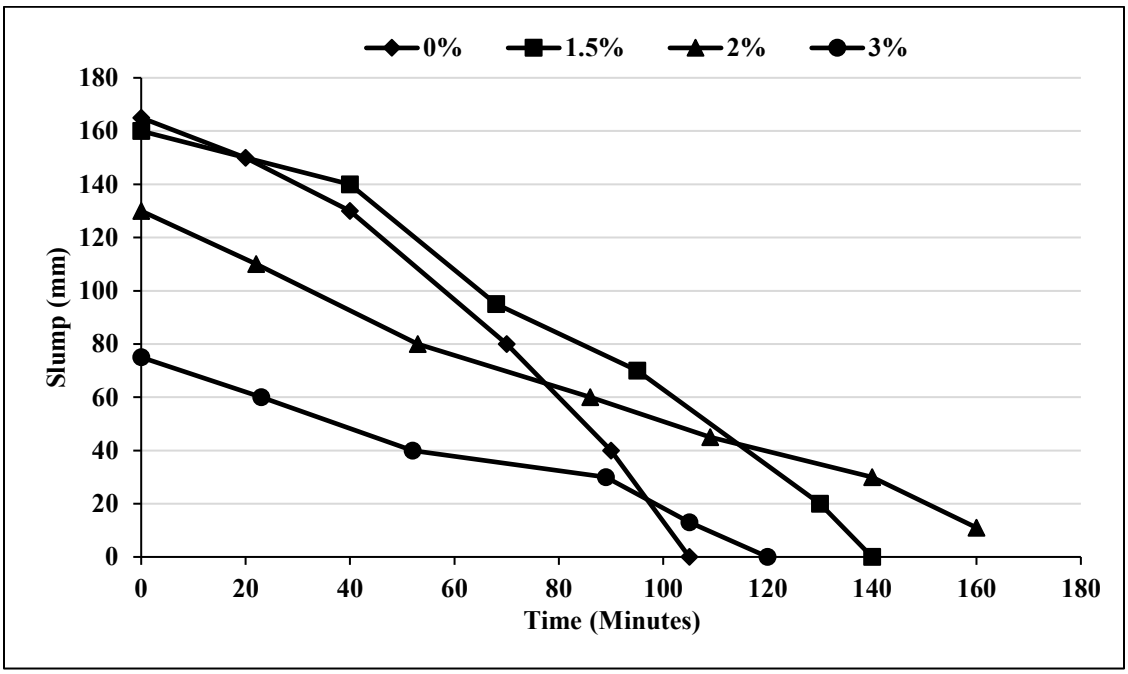

Figure 4.20: Slump Retention in Concrete Mixes with Varying Dosage of Oil Ash.

Table 4.17 shows initial slump values and the time required for concrete to lose its workability upon using oil ash.

Table 4.17: Effect of Oil Ash on Slump Retention in Concrete.

Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
0%	165	105
1.5%	160	140
2%	130	160
3%	75	120

According to the data in Table 4.17, the addition of 1.5%, 2% and 3% oil ash reduced the initial slump values in concrete in comparison to the case of 0% addition, due to the increase in the content of fine materials in the mix since oil ash has extremely fine particles. The use of 1.5, 2 and 3% oil ash improved the slump retention time, and the improvement was maximum at 2% oil ash.

4.4.4 Effect of Commercial Retarders on Slump Retention

The variation of slump with time in concrete mixes incorporating the commercial retarders Conplast RP264® and Sika Retarder 21® is plotted in Figure 4.21

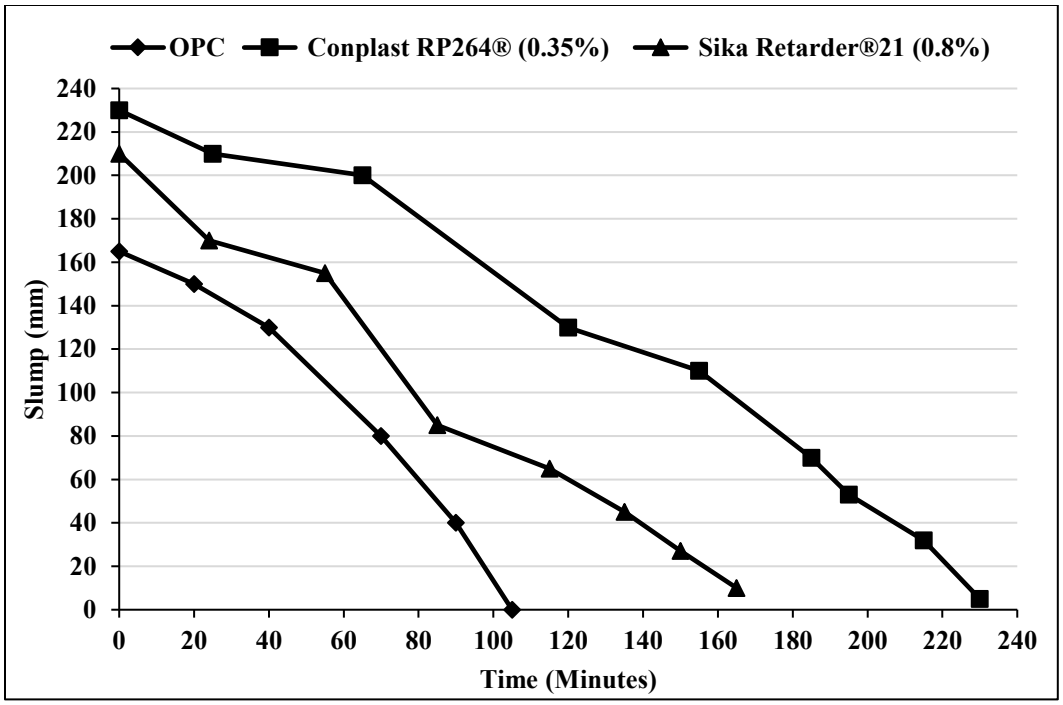

Figure 4.21: Slump Retention in Concrete Mixes Containing Commercial Retarders.

Table 4.18 presents the initial slump values and slump retention time in concrete containing commercial retarders Conplast RP264® and Sika Retarder 21®.

Table 4.18: Effect of Commercial Retarders on Slump Retention in Concrete.

Material	Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
OPC	--	165	105
Conplast RP264®	0.35%	230	170
Sika Retarder 21®	0.80%	210	230

The addition of 0.35% Conplast RP264® and 0.8% Sika Retarder 21® improved the slump retention time. However, the effect was stronger by using Conplast RP264®. The improvement in slump retention is due to the retardation influence of the admixtures.

4.4.5 Effect of Chemical Retarders on Slump Retention

The influence of incorporating sugar, ZnO and sodium gluconate on the variation of slump with time in concrete is presented in Figure 4.22.

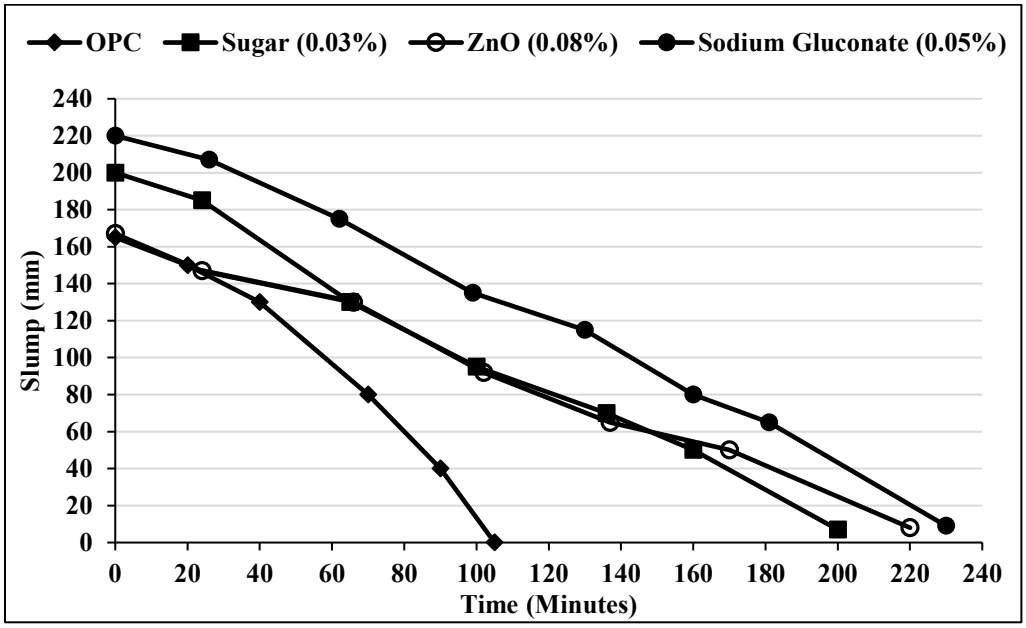

Figure 4.22: Slump Retention in Concrete Mixes Containing Chemical Retarders.

Table 4.19 presents the effect of sugar, ZnO and sodium gluconate on initial slump and slump retention time in concrete.

Table 4.19: Effect of Chemical Retarders on Slump Retention in Concrete.

Material	Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
OPC	--	165	105
Sugar	0.03%	200	200
ZnO	0.08%	167	220
Sodium Gluconate	0.05%	220	230

According to the data in Table 4.19, the addition of sugar and zinc oxide significantly increased the initial slump and improved the slump retention time in concrete. However, sodium gluconate was the most effective in the improvement of slump retention time.

Workability classification according to the European Standard BS EN 206-1 [73] is given in Table 4.20. The classification is a scale in which class S1 represents the least workable concrete which requires tamping compaction, for example, concrete used in kerb bedding and backing. Class S4 represents the most workable concrete (self-compacting concrete) which is used in applications such as in-situ piling. Table 4.20 is to be used to assess the workability of each mix as affected by retarders.

Table 4.20: Classification of Workability According to BS EN 206-1.

Classification of Workability	Form of Compaction	Slump (mm)
S1	Tamping	10-40
S2	Poker or beam vibration	50-90
S3	Tamping and/or Poker or beam vibration	100-150
S4	Self-weight compaction	⩾160

Table 4.21 presents a summary of the effects of retarders on slump retention time in concrete. All additions of retarders enhanced the initial slump values in concrete, except oil ash and thermally treated EAFD since they increase water requirements of the mix. The initial slump values of concrete mixes containing EAFD and ZnO were between 167 mm and 175 mm, so concrete is classified as self-compacting according to BS EN 206-1. Furthermore, Conplast RP264®, Sika Retarder21®, sugar and sodium gluconate produced initial slumps of more than 200 mm in concrete, which is also classified as self-compacting.

The time required for plain concrete to lose its workability was 105 minutes. The addition of 2 and 3% EAFD raised that time up to 180 and 205 minutes, respectively. The use of the commercial and chemical retarders produced slump retention time between 170 minutes and 230 minutes. Slump retention time was between 120 minutes and 175 minutes after the addition of T-EAFD and oil ash.

Table 4.21:Effect of Retarders on Slump Retention in Concrete.

Material	Dosage	Initial Slump (mm)	Slump Retention Time (Minutes)
OPC	--	165	105
EAFD	0.5%	167	145
	1%	170	155
	2%	173	180
	3%	175	205
T-EAFD	30%	118	140
	40%	75	175
Oil Ash	1.5%	160	140
	2%	130	160
	3%	75	120
Conplast RP264®	0.35%	230	230
Sika Retarder21®	0.80%	210	170
Sugar	0.03%	200	200
ZnO	0.08%	167	220
Sodium Gluconate	0.05%	220	230

4.5 COMPRESSIVE STRENGTH DEVELOPMENT

4.5.1 Effect of EAFD on Compressive Strength Development

Compressive strength development in concrete which contains different dosages of EAFD is depicted in Figure 4.23. The compressive strength of concrete increases with the time of curing. At earlier ages, the addition of EAFD reduced the compressive strength. The reduction in strength was expected because of the retardation influence of EAFD. After 28 days, the average compressive strength in concrete with 0, 0.5, 1, 2, and 3% EAFD was 48.36, 48.83, 49.25, 49.90 and 50.71 MPa, respectively.

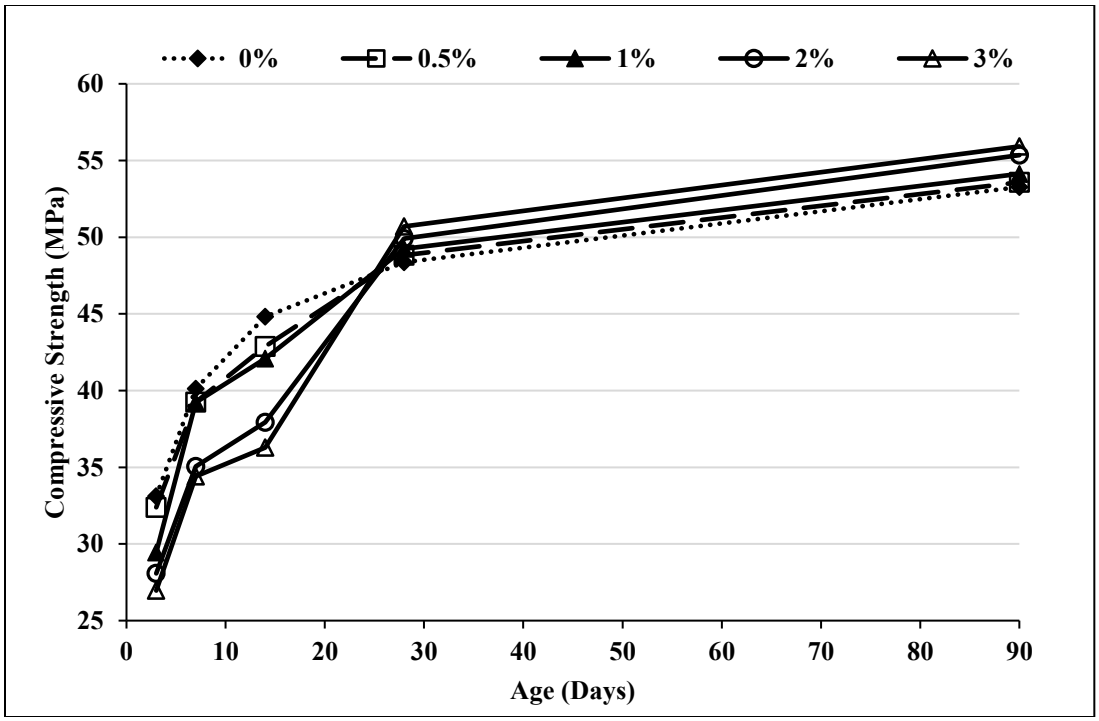

Figure 4.23: Compressive Strength of Concrete with Varying Dosage of EAFD.

4.5.2 Effect of Thermally Treated EAFD on Compressive Strength Development

According to Figure 4.24, the compressive strength of concrete increases with the time of curing. The early-age compressive strength given by specimens incorporating T-EAFD was close to that of plain concrete. After 28 days, the average compressive strength of 0, 30 and 40% T-EAFD concrete was 48.36, 50.77 and 51.73 MPa, respectively. After 90 days, the average compressive strength was 53.27, 53.75 and 53.91 MPa, respectively.

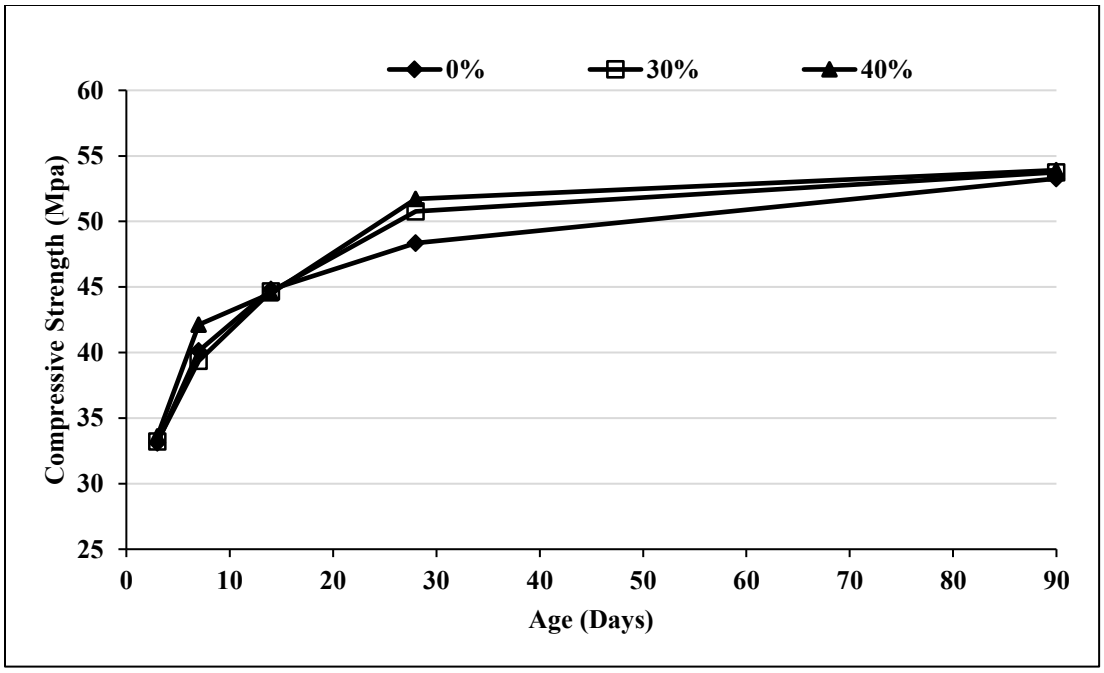

Figure 4.24: Compressive Strength of Concrete with Varying Dosage of T-EAFD.

4.5.3 Effect of Oil Ash on Compressive Strength Development

As shown in Figure 4.25, which presents the development of compressive strength of concrete after oil ash addition, oil ash reduced the compressive strength of concrete as it retards the hydration of cement. After 28 days, the average compressive strength of concrete containing 0, 1.5, 2 and 3% oil ash was 48.36, 49.85, 51.07 and 52.09 MPa, respectively.

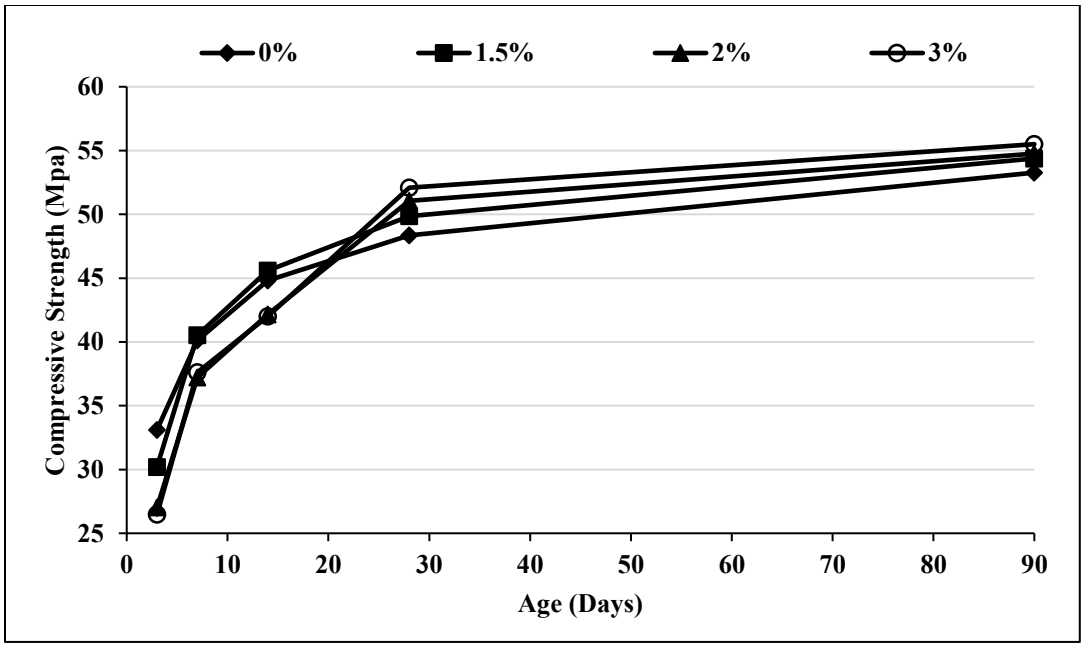

Figure 4.25: Compressive Strength of Concrete with Varying Dosage of Oil Ash.

4.5.4 Effect of Commercial Retarders on Compressive Strength Development

The addition of Conplast RP264® and Sika Retarder 21® decreased the early-age compressive strength of concrete that is anticipated. After 28 days of curing, the average compressive strength of concrete containing 0.35% Conplast RP264 and 0.8% Sika Retarder 21 was 48.53 and 46.45 MPa, respectively. The 90-day compressive strength of plain OPC concrete was 48.36 MPa, as shown in Figure 4.26.

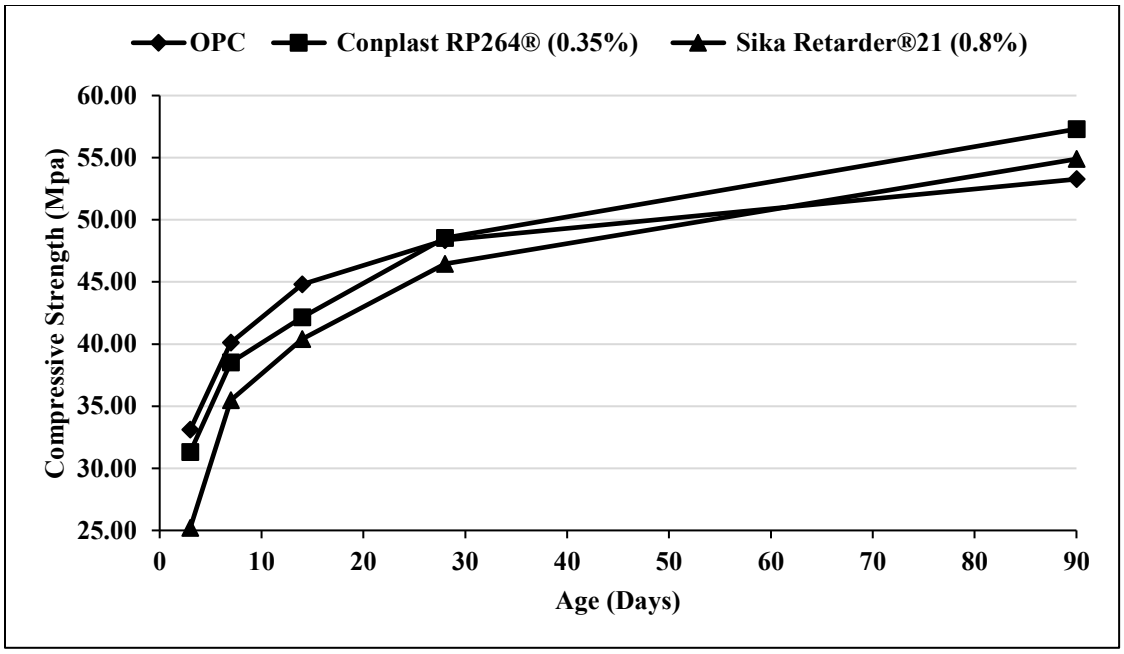

Figure 4.26: Compressive Strength of Concrete Containing Commercial Retarders.

4.5.5 Effect of Chemical Retarders on Compressive Strength Development

Influence of chemical retarders on the development of compressive strength of concrete is plotted in Figure 4.27. Up to 7 days, the compressive strength of concrete containing 0.08% ZnO was slightly lower than that of plain concrete. Both 0.03% sugar and 0.05% sodium gluconate also reduced the early-age compressive strength. The 28-day compressive strength of concrete containing 0.03% sugar, 0.08% ZnO and 0.05% sodium gluconate was 48.51, 52.61 and 49.60 MPa, respectively.

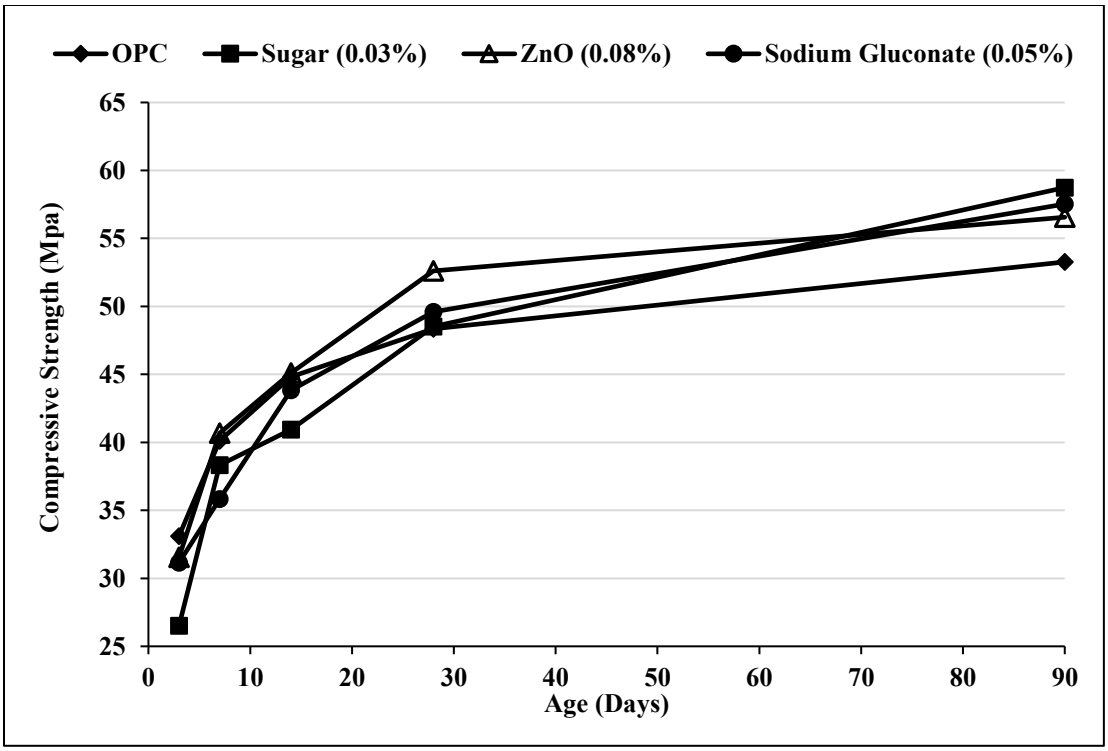

Figure 4.27: Compressive Strength of Concrete Containing Chemical Retarders.

The average compressive strength of concrete tested after 3, 7, 14, 28, and 90 days of water curing is summarized in Table 4.22. The results show that all the retarding materials reduced the 3-day compressive strength except thermally treated EAFD which gave a 3-day compressive strength slightly higher than that of plain concrete. The reduction in the 3-day compressive strength was more noted in EAFD, oil ash, Sika Retarder 21® and sugar. After 28-days of water curing, all the retarders enhanced the compressive strength except Sika Retarder 21® which is still causing a slight reduction in the compressive strength (about 4% reduction). After 90 days of curing, each concrete mix containing a retarder gave an enhanced compressive strength. However, the level of enhancement varied among the different materials. The addition of 0.03% sugar enhanced the compressive strength by 10.3%, which is the maximum enhancement in all mixes.

Table 4.22: Average Compressive Strength of Concrete Mixes.

Material	Dosage	Average Compressive Strength (MPa)				
		Day 3	Day 7	Day 14	Day 28	Day 90
OPC	--	33.11	40.12	44.81	48.36	53.27
EAFD	0.5%	32.39	39.24	42.88	48.83	53.58
	1%	29.45	39.22	42.09	49.25	54.13
	2%	28.08	35.05	37.93	49.90	55.35
	3%	26.96	34.41	36.28	50.71	55.92
T-EAFD	30%	33.21	39.37	44.66	50.77	53.75
	40%	33.62	42.12	44.53	51.73	53.91
Oil Ash	1.5%	30.20	40.52	45.58	49.85	54.38
	2%	27.02	37.21	42.16	51.07	54.76
	3%	26.47	37.61	42.00	52.09	55.50
Conplast RP264®	0.35%	31.31	38.52	42.16	48.53	57.30
Sika Retarder21®	0.80%	25.20	35.47	40.41	46.45	54.89
Sugar	0.03%	26.50	38.33	40.94	48.51	58.74
ZnO	0.08%	31.54	40.67	45.10	52.61	56.56
Sodium Gluconate	0.05%	31.15	35.83	43.83	49.60	57.35

4.6 DRYING SHRINKAGE

4.6.1 Effect of EAFD on Drying Shrinkage

Evolution of the drying shrinkage of mortars incorporating EAFD is presented in Figure 4.28. It is apparent that the drying shrinkage of mortars increases in response to the increase in the duration of air drying. The figure also shows an initial, rapid increase in the drying shrinkage over the first few days, followed by a slower increase in the shrinkage over later ages. The addition of EAFD caused an increase in the drying shrinkage. After 90 days in air, the drying shrinkage strain of mortar containing 0, 0.5, 1, 2 and 3% EAFD was 861, 864, 867, 986 and 1153 microns, respectively.

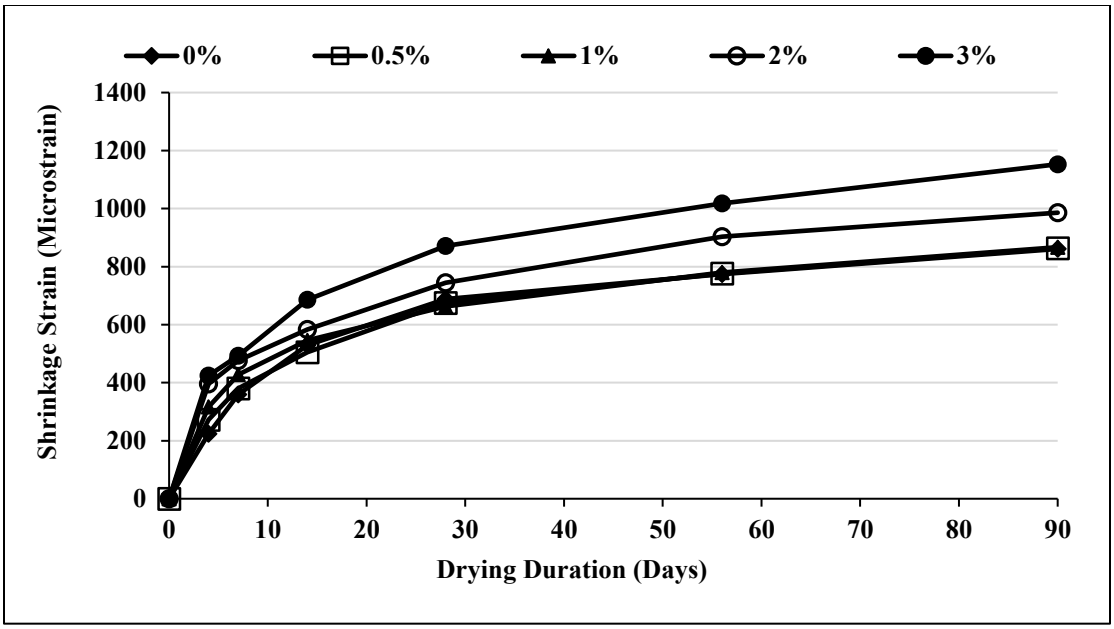

Figure 4.28: Drying Shrinkage of Mortar with Varying Dosage of EAFD.

4.6.2 Effect of Thermally Treated EAFD on Drying Shrinkage

The increase of drying shrinkage in mortar specimens, which contain T-EAFD, is depicted in Figure 4.29. The drying shrinkage increased with duration of air storage, and the addition of T-EAFD caused a further increase in the drying shrinkage. After 90 days, drying shrinkage strain of 861, 1329 and 1498 microns was observed in 0, 30 and 40% T-EAFD mortar, respectively.

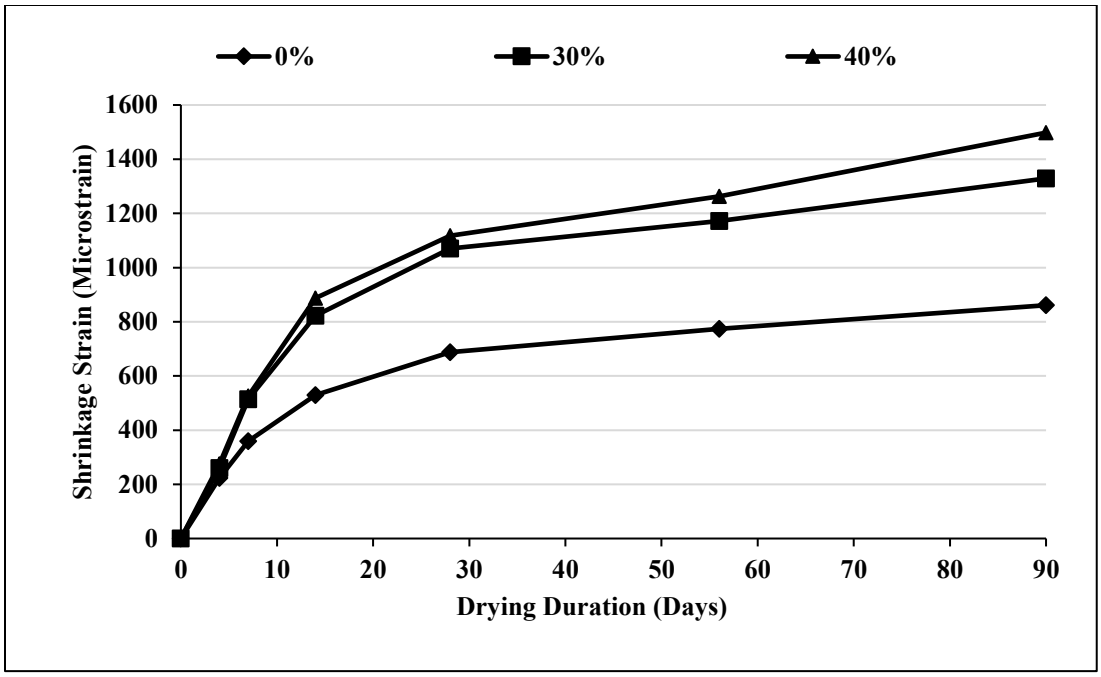

Figure 4.29: Drying Shrinkage of Mortar with Varying Dosage of T-EAFD.

4.6.3 Effect of Oil Ash on Drying Shrinkage

As shown in Figure 4.30, oil ash caused an increase in the drying shrinkage with a rapid rate at the first few days. Thereafter, the rate of increase of drying shrinkage slowed down. After 90 days, the drying shrinkage strain of mortar containing 0, 1.5, 2 and 3% oil ash was 861, 1005, 996 and 947 microns, respectively.

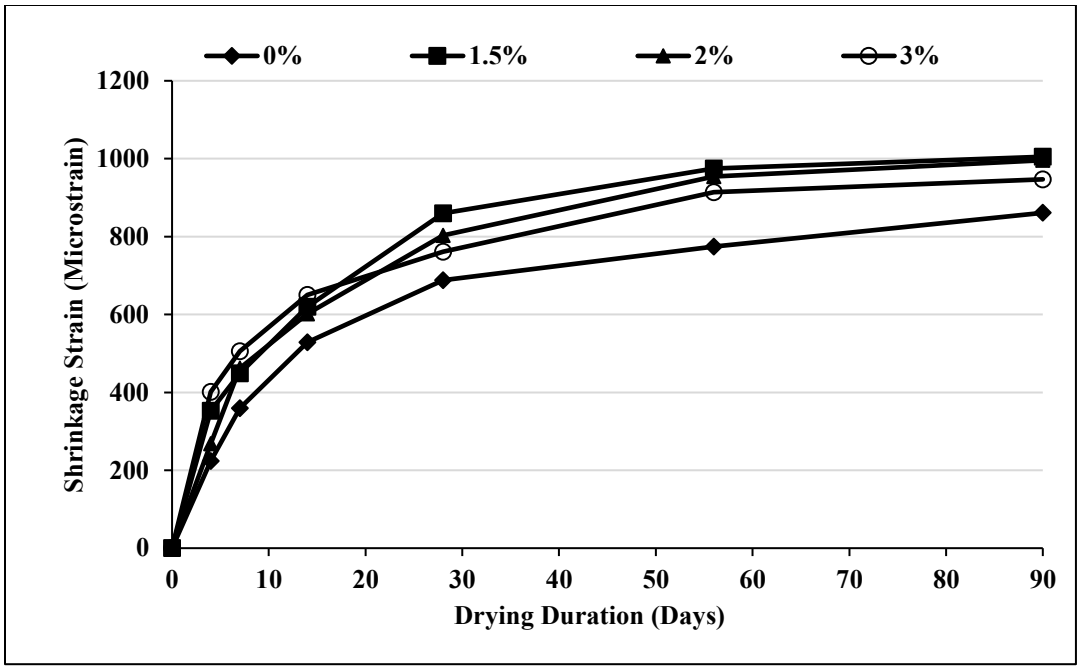

Figure 4.30: Drying Shrinkage of Mortar with Varying Dosage of Oil Ash.

4.6.4 Effect of Commercial Retarders on Drying Shrinkage

The evolution of the drying shrinkage in mortar specimens that contain Conplast RP264 and Sika Retarder 21 is plotted in Figure 4.31. The drying shrinkage increased with duration of air storage. Moreover, the addition of both commercial retarders caused a further increase in the drying shrinkage. After 90 days, a drying shrinkage strain of 1036 and 990 microns was measured in the mix incorporating 0.35% Conplast RP264 and 0.8% Sika Retarder 21, respectively.

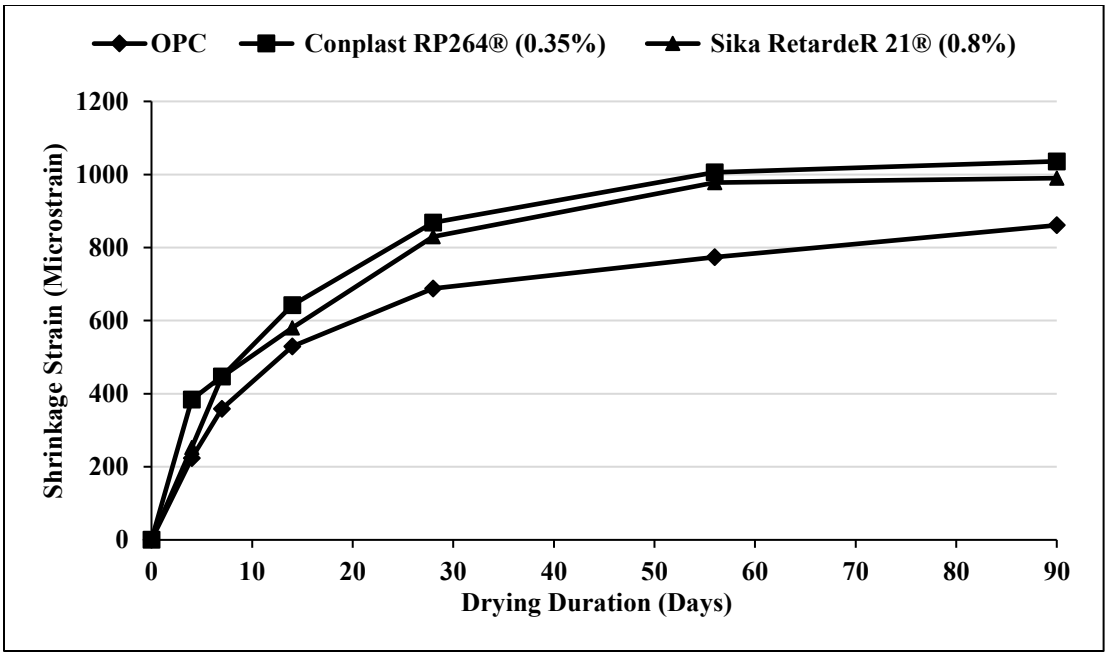

Figure 4.31: Drying Shrinkage of Mortar Containing Commercial Retarders.

4.6.5 Effect of Chemical Retarders on Drying Shrinkage

Figure 4.32 presents the influence of chemical retarders on the drying shrinkage of mortar. The figure shows that the drying shrinkage increases with time. Over the first few days, the rate of increase of drying shrinkage was rapid. Thereafter, it started to slow down. The use of chemical retarders caused an increase in the drying shrinkage of mortar. After 90 days, the drying shrinkage strain of plain OPC mortar was 861 microns. However, this value was increased to 1027, 1018 and 903 microns with the use 0.03% sugar, 0.08% ZnO and 0.05% sodium gluconate, respectively.

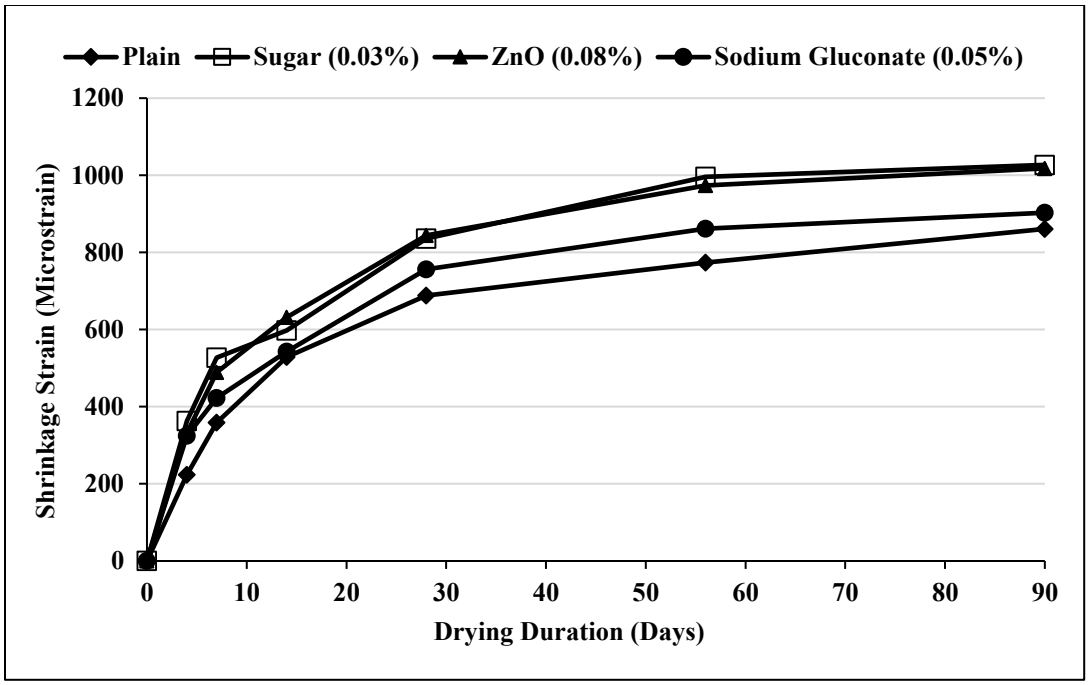

Figure 4.32: Drying Shrinkage of Mortar Containing Chemical Retarders.

The drying shrinkage was measured in mortar containing the retarding materials during the first 90 days following water curing. All drying shrinkage readings are summarized in Table 4.23.

According to the ACI, the drying shrinkage strain should be less than 500 microns at the age of 7 days. As shown in Table 4.23, the drying shrinkage strain of plain OPC mortar after 7 days of air drying was 359 microns which is the lowest among all mortar. This value was increased due to the addition of retarders. Incorporation of 3% EAFD resulted in a drying shrinkage of 493 microns. Moreover, the drying shrinkage in the mortar with 0.35% Conplast RP264® and 0.8% Sika Retarder21® was 447 and 446 microns, respectively. However, 40% thermally treated EAFD, 3% oil ash and 0.03% sugar induced a drying shrinkage strain of more than 500 microns, which exceeds the ACI limit. Thus, precautionary measures are to be adopted to prevent any consequences. The drying

shrinkage of mortar containing ZnO and sodium gluconate was less than 500 microns at the age of 7 days.

After 90 days, the minimum drying shrinkage was observed in plain OPC mortar (861 micron). The maximum drying shrinkage was measured in 40% thermally treated EAFD, the value was 1498 microns, which can be justified by the retardation effect and the increase in the amount of fines in the mortar.

Table 4.23: Drying Shrinkage Strain of Mortar.

Material	Dosage	Drying Shrinkage (Microns)					
		Day 4	Day 7	Day 14	Day 28	Day 56	Day 90
OPC	--	224	359	529	688	774	861
EAFD	0.5%	272	381	505	675	777	864
	1%	317	428	546	663	780	867
	2%	396	476	583	744	903	986
	3%	425	493	686	871	1018	1153
T-EAFD	30%	260	514	822	1070	1172	1329
	40%	276	526	887	1118	1263	1498
Oil Ash	1.5%	353	449	620	860	975	1005
	2%	268	462	602	803	954	996
	3%	401	506	650	761	914	947
Conplast RP264®	0.35%	384	447	642	868	1006	1036
Sika Retarder21®	0.80%	253	446	580	830	978	990
Sugar	0.03%	363	527	598	836	987	1027
ZnO	0.08%	332	489	632	844	974	1018
Sodium Gluconate	0.05%	325	423	543	756	862	903

4.7 COMPARISON OF RETARDERS

Table 4.24 presents a summary for the effects of the studied retarders on different properties of cement paste, mortar and concrete including setting time, slump retention, compressive strength and drying shrinkage.

Table 4.24: Effects of Retarders on Setting Time, Slump Retention, Compressive Strength and Drying Shrinkage.

Material	Dosage	IST (Minutes)	FST (Minutes)	Initial Slump (mm)	Slump Retention Time (Minutes)	28-Day Compressive Strength (MPa)	7-Day Drying Shrinkage (Microns)
OPC	--	140	205	165	105	48.36	359
EAFD	0.50%	165	225	167	145	48.83	381
	1%	180	240	170	155	49.25	428
	2%	280	315	173	180	49.9	476
	3%	440	465	175	205	50.71	493
T-EAFD	30%	180	220	118	140	50.77	514
	40%	205	260	75	175	51.73	526
Oil Ash	1.50%	180	245	160	140	49.85	449
	2%	210	290	130	160	51.07	462
	3%	235	315	75	120	52.09	506
Conplast RP264®	0.35%	435	450	230	230	48.53	447
Sika Retarder 21®	0.80%	450	495	210	170	46.45	446
Sugar	0.03%	370	395	200	200	48.51	527
ZnO	0.08%	335	430	167	220	52.61	489
Sodium Gluconate	0.05%	215	275	220	230	49.6	409

As shown in Table 4.24, the initial and final setting time varied according to the material type and dosage. Thus, a certain required initial setting time can be attained by selecting the material and its dosage. EAFD, commercial retarders, sugar and zinc oxide can be used to achieve initial setting time of more than 300 minutes. Oil ash can be used to extend the initial setting time up to 235 minutes, and thermally treated EAFD can prolong the initial setting time up to 205 minutes. Sodium gluconate can be used to obtain an initial setting time of 215 minutes. Workability of concrete was also affected by retarders. EAFD increased the initial slump, it also extended the slump retention time up to 205 minutes. Commercial and chemical retarders also increased the initial slump. The slump retention

time was in the range of 170 minutes to 230 minutes. Oil ash and thermally treated EAFD reduced the initial slum but they extended the slump retention time. The 28-day compressive strength was not significantly affected by any retarders. The use of retarders increased the drying shrinkage. However, the 7-day drying shrinkage strain was less than 500 microns except for 30 and 40% EAFD, 3% oil ash and 0.03% sugar. The 7-day drying shrinkage was maximum due to 0.03%sugar at 527 microns.

4.8 SCANNING ELECTRON MICROSCOPY (SEM)

To evaluate the influence of the retarders on the microstructure of cement paste, some samples were selected to be analyzed using SEM. The selected samples include both plain cement paste and paste containing 3% EAFD, 40% T-EAFD, 3% oil ash, 0.35% Conplast RP264® and 0.80% Sika Retarder 21®. The SEM was conducted on the selected paste specimens that were cured for 28 days in water.

Figure 4.33 through Figure 4.38 show SEM pictures of all selected specimens. Plain cement paste showed a paste of normal morphology with a dense structure (Figure 4.33). The addition of 3% EAFD resulted in a paste of normal morphology but with less dense structure compared to plain cement paste as shown in Figure 4.34. Paste containing 40% T-EAFD and paste containing 3% oil ash have a normal morphology but with higher porosity compared to plain cement paste (Figures 4.35 and 4.36). Paste containing 0.35% Conplast RP264® has a less dense morphology as presented in Figure 4.37. The use of 0.80% Sika Retarder 21® did not affect the normal morphology of cement paste. However, it resulted in a paste with less dense structure compared to plain cement paste (Figure 4.38). Neither carbonation nor micro cracks were observed in any of the samples. It can be

concluded that the use of retarders generally increases the porosity and reduces the density of the microstructure of cement paste which might affect the durability characteristics of the concrete.

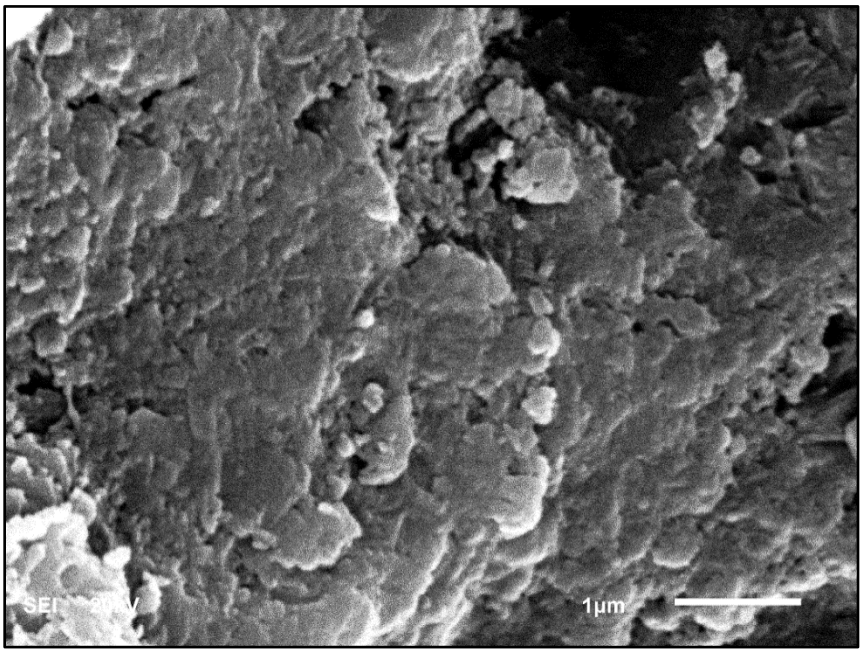

Figure 4.33: SEM for Plain Cement Paste.

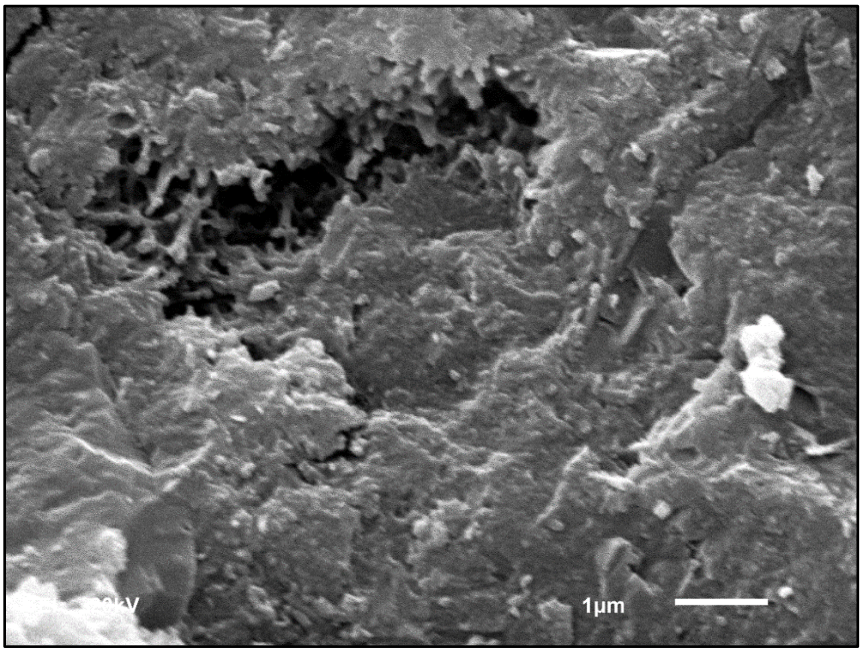

Figure 4.34: SEM for Paste Containing 3% EAFD.

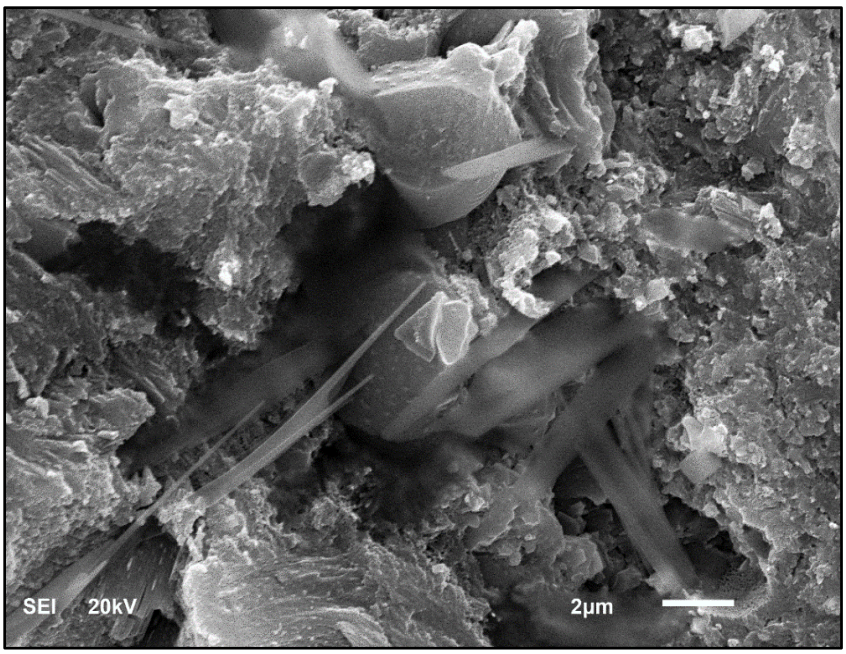

Figure 4.35: SEM for Paste Containing 40% Thermally Treated EAFD.

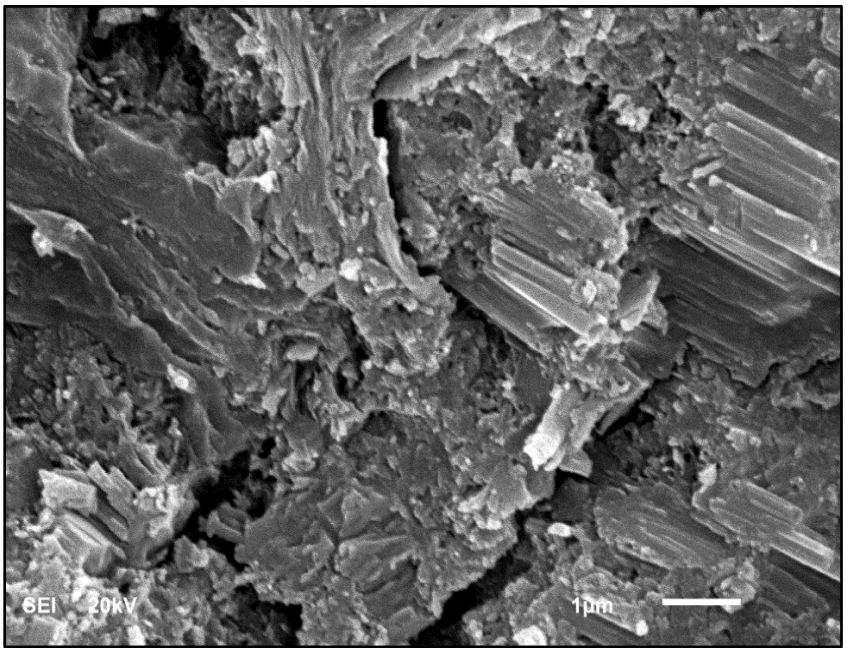

Figure 4.36: SEM for Paste Containing 3% Oil Ash.

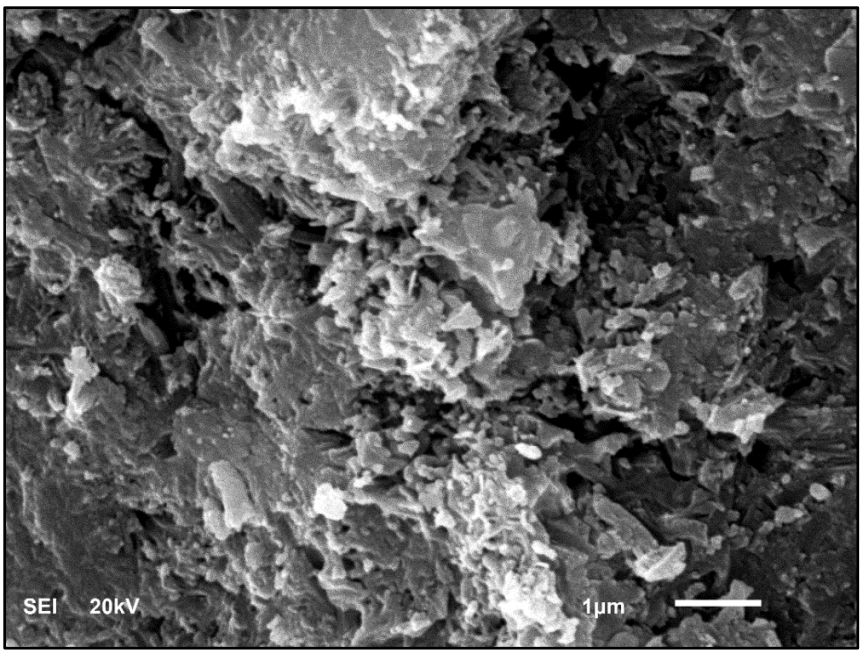

Figure 4.37: SEM for Paste Containing Conplast RP264® (0.35%).

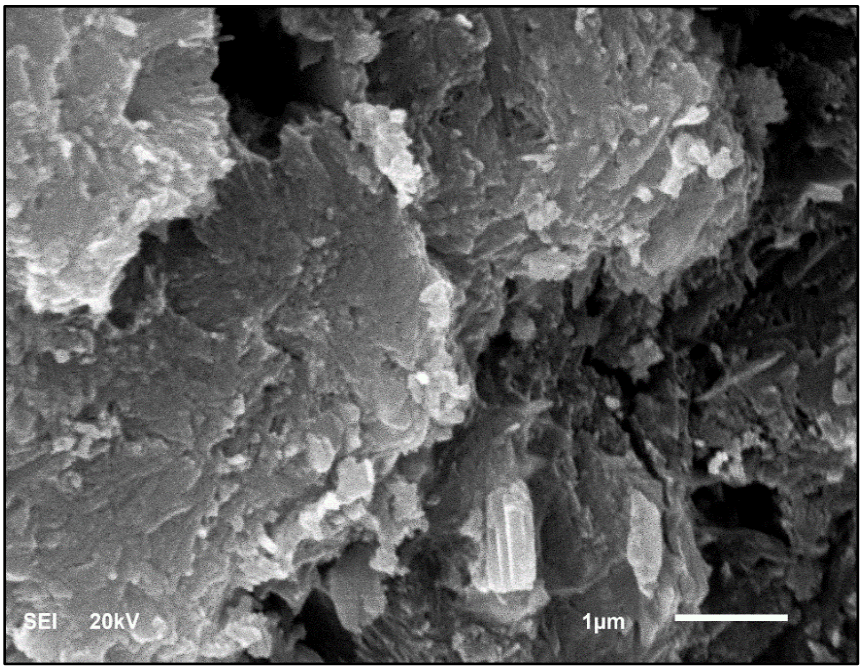

Figure 4.38: SEM for Paste Containing Sika Retarder 21® (0.80%).

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

This research was conducted to develop sustainable concrete set retarders using indigenous industrial byproducts. After the analysis and interpretation of experimental data, the following conclusions are made:

1. The use of up to 3% EAFD increased the initial setting time of OPC from 140 minutes to 440 minutes; it also increased the final setting time from 205 minutes to 465 minutes. The use of 3% EAFD increased the retention time of 80-mm slump from 70 minutes to 95 minutes. All dosages of EAFD enhanced the compressive strength and increased the drying shrinkage. However, the 7-day drying shrinkage was less than the threshold value of 500 microns.
2. The heat treatment of EAFD at 800 °C for 2 hours reduced its retardation of efficiency. However, the use of 30% and 40% additions increased the initial setting time up to 180 and 205 minutes, respectively, while the final setting time was increased up to 220 and 260 minutes, respectively. The thermal treatment of EAFD slightly enhanced the compressive strength, but it reduced the workability and increased the 7-day drying shrinkage to more than 500 microns, the threshold limit.
3. The addition of up to 3% oil ash increased the initial setting time and the final setting time of cement to 235 and 315 minutes, respectively. The compressive

strength of concrete incorporating oil ash was comparable to that of concrete without retarder. However, the water requirements increased, and thus workability was reduced. The drying shrinkage was less than 500 microns for mortar with 1.5 and 2% oil ash, while it was more than 506 microns in the mortar with 3% oil ash.

4. The initial setting time and the final setting time were 435 minutes and 450 minutes, respectively, in cement paste with 0.35% Conplast RP264®. There was no adverse effect on the compressive strength of concrete and drying shrinkage of mortar incorporating Conplast RP264®. Addition of 0.35% Conplast RP264® increased the retention time of 80-mm slump from 70 minutes to 177 minutes.
5. The initial setting time and the final setting time were 450 and 495 minutes, respectively, in cement paste with 0.8% Sika Retarder 21®. The compressive strength was slightly reduced after 28 days of curing. However, the 90-day compressive strength was similar to the of concrete without retarder. The retention time of 80-mm slump was around 90 minutes compared to 70 minutes in plain concrete. The drying shrinkage after 7 days was 446 microns that is less than the threshold value of 500 microns.
6. The addition of 0.03% sugar increased the initial setting time and the final setting time up to 370 and 395 minutes, respectively. The use of 0.03% sugar retained the slump of 80 mm for about 120 minutes. However, the drying shrinkage after 7 days was 527 microns that is more than the threshold value of 500 microns. The addition of sugar (0.03%) enhanced the 90-day compressive strength by 10.3%.
7. The addition of 0.8% zinc oxide increased the initial setting time and the final setting time to 335 and 430 minutes, respectively. Also, the 28-day compressive

strength increased up to this dosage. The 80-mm slump retention time was 117 minutes, and no adverse effect on drying shrinkage was observed.

8. Sodium gluconate was less effective in prolonging the initial setting time. A maximum initial setting time of 215 minutes was achieved by the addition of 0.05%. However, addition of 0.05% sodium gluconate increased the 80-mm slump retention time to 160 minutes. The 7-day drying shrinkage was 406 microns, and the compressive strength was slightly more than that of concrete without retarder.
9. It can be concluded that different retarders have different effects on setting time and slump retention. Generally, no adverse effects on the later-age compressive strength were observed when the final setting time was less than 500 minutes. However, the selected set retarders increased the drying shrinkage.
10. There was a good correlation between the initial and final setting time and the evolution of heat of hydration; therefore, the results of heat of hydration tests can be used to evaluate the setting time.
11. The use of the industrial wastes (EAFD and oil ash) as retarders in concrete provides several technical advantages which would bring positive environmental and economic impact.

5.2 RECOMMENDATIONS

Based on initial setting time results, different dosages of retarders were recommended as presented in Table 5.1.

Table 5.1: Recommended Dosages of Retarders Based on Initial Setting Time.

Initial Setting Time (Minutes)				
Up to 200	200-260	260-320	320-380	380-440
Conplast RP264® (0.08%)	Sugar (0.02%)	Conplast RP264® (0.20%)	EAFD (2.5%)	Sika Retarder 21® (0.80%)
Sugar (0.01%)	EAFD (1.5%)	Sika Retarder 21® (0.44%)	Conplast RP264® (0.28%)	EAFD (3%)
Sika Retarder 21® (0.20%)	Conplast RP264® (0.12%)	ZnO (0.05%)	Sika Retarder 21® (0.57%)	Conplast RP264® (0.35%)
Sodium Gluconate (0.02%)	Sika Retarder 21® (0.30%)	EAFD (2%)	ZnO (0.08%)	--
EAFD (1%)	Sodium Gluconate (0.05%)	--	--	--
Oil Ash (1.5%)	Oil Ash (2%)	--	--	--
Oil Ash (1%)	ZnO (0.02%)	--	--	--
ZnO (0.01%)	--	--	--	--
EAFD (0.5%)	--	--	--	--
T-EAFD (10%)	--	--	--	--

Based on slump retention time, different dosages of retarders were recommended as presented in Table 5.2.

Table 5.2: Recommended Dosages of Retarders Based on Slump Retention Time.

Slump Retention Time (Minutes)			
Up to 140	145 -170	175 – 200	205 - 230
Oil Ash (1.5%)	Sika Retarder 21® (0.8%)	EAFD (2%)	Conplast RP264® (0.35%)
	Oil Ash (2%)		Sodium Gluconate (0.05%)
	EAFD (1%)		ZnO (0.08%)
	EAFD (0.5%)		EAFD (3%)

5.3 SUGGESTIONS FOR FUTURE RESEARCH

1. The performance of retarders under field conditions, such as elevated temperature, humidity, and high wind speed is recommended to be studied.

2. Other mechanical and durability aspects of concrete with selected retarders need to be investigated.

3. Compatibility between commercial retarders, solid retarders and supplementary cementing materials needs to be investigated.

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